DESIGN AND EVALUATION OF ACRYLIC-BASED GROUTS FOR EARTHEN PLASTERS

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I am I plus my surroundings and if I do not preserve the latter, I do not preserve myself.
-Jose Ortega Y Gasset [Meditations on Quixote] (1883-1955)

All knowledge and discovery is built upon that which precedes it and all current conditions both tangible and intangible, are evidence of this principle of interdependent nature. Conservation embodies these principles of causation and interdependency in its embrace and integration of multiple disciplines. It recognises the inherent relationships between the extrinsic and the intrinsic, between the physical and the immaterial. It realises these principles in the thoughtful practice of its etiological and hermeneutic investigations. The philosophy and theory provide the framework for the praxis. This perspective and ultimate material contribution are manifested in the extension of the life of the 'thing,' (object, structure, site, place). This act is founded on the belief that the past and context are profound agents of who and what we are and what we envision ourselves to be. Like the quote by Jose Ortega Y Gasset acknowledges, we do not exist alone as singular, contained entities but rather are creations of a continuum.

This work is a result of that continuum. Special recognition is due to those who provided generous contributions of advice, guidance and materials. The research was sponsored by the University of Pennsylvania and the United States National Park Service.

The inexpressibility of one's gratitude is most keenly felt when attempting to articulate the indebtedness to one's teachers. A special thanks is due to the irrepayable generosity and

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And none of this would have been possible without the love and encouragement of my friends and family. I would like to thank them for their boundless belief, support and confidence, especially my Grandmother, my mother and Annalisa.

Kecia L. Fong
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CONTENTS

LIST OF FIGURES

LIST OF TABLES

PREFACE

The United States National Park Service (NPS) is responsible for a significant collection of our nation's architectural heritage. Classified as sites, monuments, and parks, these structures exist primarily out of doors in uncontrolled environments. In an agreement initiated in 1991, the National Park Service and the University of Pennsylvania embarked on what is now an eight-year cooperative research and training agreement in the conservation of historic structures and sites in the American Southwest. Much of the architecture of this region is built of earth, as it was the most available and renewable resource. Adobe blocks, puddled earth, and earthen mortars and plasters are some of the many forms in which this pliable and expressive material has been used. Maintenance is an inherent activity in the renewal and continuation of any structure, culture, and tradition. Now belonging to the public domain, these 'properties' are no longer inhabited. As such, the cycles and activity of maintenance have taken on a different pace and significance, no longer an act of distinct cultural continuity, the lifespan demands of these maintenance actions present greater challenges. What was once renewed on an annual or use-related basis, such as the act of plastering, must now be preserved in a primarily *as is* state. The challenge is how to prolong the life of buildings or sites that are now divorced from their original context. The history of these structures is renewed through the act of visitor interpretation multiple times each day, but the tangible forms are engaged in an irreversible cycle of existence. The conservation programme does not attempt to restore the physical forms, but rather to preserve and extend the life of these forms towards an enhanced comprehension of the physical fabric as an inextricable component of the

whole - site, place, history. Together, the NPS and the University of Pennsylvania are joined in their commitment for the care of this invaluable cultural heritage.

The research of this project is a result of this cooperative agreement and shared interest. Its case study site, Casa Grande Ruins National Monument is America's first nationally designated prehistoric reserve. Its 'Great House' was first stabilized by Cosmos Mindeleff in 1892 with 'public funds expended by an appropriation from Congress.'[1] The National Park Service assumed management of the site in 1917, and so began a long and continuing tradition of preservation.

The issues of heritage management are multi-faceted and constantly changing. These changes in perspectives will always reflect the cultural, social and political concerns of the moment. The material issues of conservation are perhaps less socially respondent, matter deteriorates and is transformed in an endless cycle. Weather will always contribute to this process for outdoor structures and unless maintained, they will deteriorate. Change is immutable. It is our perspective, approach, knowledge, and technology that are mutable.

Originally functioning as renewable architectural elements and dispensable in past preservation efforts, surface finishes are now regarded as integral components of the architectural composition. They are the skins of a structure, often the last application in construction and, the first thing to alter through weathering. Surface finishes often carry the most detailed messages found in the forms of painted, incised or polished applications. As the

outermost surface they offer protection to the rest and by their nature tend to be the most temporal of the architectural elements. As a protective, sacrificial coat they are frequently renewed. These multiple applications, layered one upon another, are like the pages of a book, each layer reveals a piece of the story; a change in perspective visible through changes in materials, techniques, colors, patterns, or physico-chemical transformation. We may no longer fully know what these ephemeral components signify but we have determined that they are of importance to us. Bound by history and the desire to make sense of ourselves and our surroundings we continue to leave our messages whether we are creating entirely new ones or reinterpreting the old.

[1] Jake Barrow and Frank Matero, Preface to *Acrylic-Modified Earthen Mortar*, by Robert Hartzler, (Santa Fe: United States National Park Service Department of the Interior, 1996), p. xiii.

1.0 INTRODUCTION

In the fall of 1995 a large fragment of the Casa Grande or "Great House" of Compound A of the Casa Grande Ruins National Monument fell from the base of the west exterior wall. Subsequent research would determine that this was not the first time a fragment had detached but its loss incited concern over the condition and future of this much revered structure. Still regarded as a sacred site to affiliated Native American communities such as the Pima and Tohono O'odham (previously known as Papago), Casa Grande holds an eminent place in the collective cultural conscience. Moreover, its outstanding significance is derived from the following claims:[1]

- It is the largest surviving prehistoric earthen (non-mound) building in the United States.

- It is the only surviving example of Classic Period Hohokam "Great House" architecture, which in addition, exists in an immediate village context not preserved elsewhere.

- The site is equally distinguished as the first federally designated and protected archaeological preserve (1889-1892); and it possess one of the earliest (1932) and largest twentieth century shelters erected to date.

In the fall of 1996, two separate research theses were proposed towards the continued conservation of the Great House. One entailed an investigation of the structure, its materials and weathering.[2] The other, this research, examines the interior earthen surface finishes, their current *in situ* condition and characterisation, and proposes a treatment solution for their reattachment and stabilisation. While the treatment proposal is driven by the needs of this particular site, the solution is designed to be widely applicable to a specific set of common

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[1] Frank Matero, "Documentation and Assessment of Wall Conditions, The Casa Grande, Casa Grande Ruins National Monument, Arizona," March 1999, unpublished report. Philadelphia, Office of the Graduate Program in Historic Preservation, University of Pennsylvania, p. 3, 4.

1

variables; finish plaster reattachment, and gap filling of a certain measured detachment (~3 - 5 mm).

Much has been written about materials and methods for the reattachment and stabilisation of lime plasters. As well, there is a measurable amount of literature on the preservation of earthen architecture particularly in the area of adobe amendments. However, very little has been published on the topic of the reattachment of earthen plasters and even less about the reattachment and stabilisation of earthen plasters *in situ*. A few germane studies on this topic are available in both published and unpublished form by Watson Smith, Giacomo Chiari, Constance Silver, and Paul Schwartzbaum. These carefully researched studies were used as the starting point for this report whose investigation extended into adobe amendment studies, the adhesives and concrete amendments industries, plaster and mural reattachment, grouting, and museum object adhesive studies.

Constance Silver performed extensive research into *in situ* earthen plaster reattachment with a focus on Ancestral Puebloan sites in the American Southwest in the early 1980's. She experimented with different polymer emulsion adhesives of varying concentrations. Morgan Phillips published some of the earliest innovations in the use of filled and unfilled acrylic emulsion-based grouts for lime plaster reattachment and gap filling. More recently, Frank Matero and Angelyn Bass have published on hydraulic lime-based grouts that utilize small percentages of acrylic emulsion as the system dispersant for the reattachment and stabilisation of lime plasters to earth. Each of these contributions refined a previous system and responded

² See Elisa Del Bono, "Characterization and Analysis of the Caliche Walls of the 'Great House,' Casa Grande Ruins

to its respective set of contextual variables and criteria. Phillips developed his system primarily for interior environments of lime plaster to wood lath. Matero and Bass were concerned with the issues surrounding uncontrolled exterior environments of lime plaster to earthen supports and relatively large scale voids. These studies served as models for the present research that involves elements of each respective program yet whose specific requirements could not wholly be fulfilled by either one alone.

All intervention begins with the identification of a problem. In this case, the detachment of large expanses of surface finish and intermediary finish (or leveling) layers of earthen architecture. Treatment options can only be derived from knowledge of the site materials, architectural function, and environmental factors. Through use history and environment, mechanisms of deterioration can be understood. Cultural, social, economic, political and administrative considerations comprise another layer of contextual definition. This last grouping is considered beyond the immediate scope of this research but cannot be ignored. Sound methodology is the process by which problem identification and treatment options are developed. This process is arranged in the following sequence, archival documentation, field recordation, site sampling, material characterisation, identification of the mechanisms of deterioration, definition of the conservation treatment problem, material literature research, laboratory testing programme, data interpretation, and recommendations.

National Monument, Casa Grande, Arizona," Master's thesis, University of Pennsylvania, 1999.

2.0 THE CASA GRANDE

2.1 Site Summary

Casa Grande Ruins National Monument (CGRNM) is located in Pinal County of south central Arizona within the Sonoran Desert. The climate is a subtropical desert where the majority of sparse precipitation is received from storms originating to the south during the summer monsoon season of July and August. Mean annual rainfall is 7.96 inches. Daytime temperatures frequently exceed 100° F in the summer. Winter temperatures are cooler and hover
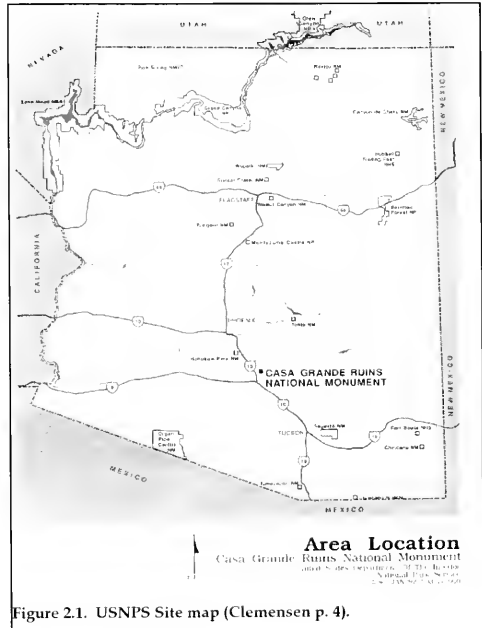
Figure 2.1. USNPS Site map (Clemensen p. 4).

around the low 80°s F by day and between 30° and 40° F by night.[1] The excavated compounds date to AD 1200-1450 and are attributed to the Hohokam culture, a Pima Indian term meaning "those who have gone."[2] The Casa Grande itself, or 'Great House' is located within compound A of the park. It is exceptional as a unique example of a massive freestanding

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[1] United States National Park Service, "Casa Grande Ruins National Monument Resources Management Plan," 1996, p. 1-10.
[2] A. Berle Clemensen , *A Centennial History of the First Prehistoric Reserve: 1892-1992*, (United States Department of the Interior/ National Park Service, 1992), p. 5.

multi-storey puddled earthen structure dating to pre-European contact.   As an architectural

remnant of the early Hohokam culture it is unmatched.
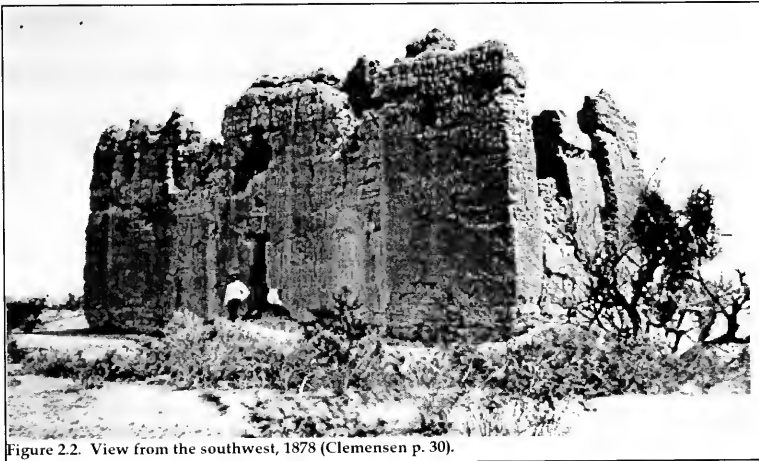
Figure 2.2.   View from the southwest, 1878 (Clemensen p. 30).

Father Eusebio Kino was the first European to record the existence of the Casa Grande in 1694.

> The Casa Grande is a four story building, as large as a castle and equal to the
> largest church in these lands of Sonora. Close to this Casa Grande there are
> thirteen smaller houses, somewhat more dilapidated, and the ruins of many
> others, which make it evident that in ancient times there had been a city here.[3]

His description drew others to the site who recorded their observations in writings, drawings,

and photographs thereby increasing its notoriety. Calls for its preservation were made as

early as 1879 and in 1887 "H. S. Jacobs, a United States Geological Survey official, advised the

Arizona people and legislature that Casa Grande had sufficient scientific and historic value

that it should be protected from vandalism and decay."[4]   Leading anthropologists and

archaeologists Adolf Bandelier, J. Walter Fewkes, and Frank Cushing each visited the site

through the 1880s and 1890s.   Bandelier submitted the first formal anthropological study of

the site.[5] Fewkes in his survey of the structure assigned letter designations to the tiers of

rooms.[6] This system of organization is still in use today. Cushing submitted an archaeological

report to the Secretary of the Interior as supporting evidence to the significance and value of

the ruin. In 1889 money was appropriated from Congress

> ...to enable the Secretary of the Interior to repair and protect the ruin of Casa
> Grande, situated in Pinal County, near Florence, Arizona, two thousand
> dollars; and the President is authorized to reserve from settlement and sale
> the land on which said ruin is situated and so much for the public land
> adjacent thereto as in his judgment may be necessary for the protection of said
> ruin and of the ancient city of which it is a part.[7]

In 1890 Victor and Cosmos Mindeleff prepared the first condition survey and list of

recommendations for the preservation of the 'Great House.' The list included the following

recommendations.[8]

1. Fence the ruins area
2. Provide a permanent on-site custodian
3. Clean the debris from the Great House
4. Underpin the Great House walls with brick
5. Remove several inches of material from the wall tops to provide a good
    bearing surface and then cap the walls with concrete
6. Reinforce the walls with tie-rods and beams, replace broken and missing
    lintels, and fill cavities above the lintels.
7. Erect a roof shelter over the structure

While not all recommendations were performed (items number two and seven would not be

realized for many years and number five has never been performed), this initial preservation

campaign represents the single largest structural intervention to date.   In 1892 President

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[4] Clemensen, *Centennial History*, p. 28-29.

[5] Clemensen, *Centennial History*, p. 29.

[6] David R. Wilcox and Lynette O. Shenk, "The Architecture of the Great House and its Interpretation," (Tucson, Arizona: United States National Park Service, 1977), p. 29.

[7] Clemensen, *Centennial History*, p. 33.

Benjamin Harrison designated 480 acres as the Casa Grande Reservation thereby establishing it as the first federal preserve set aside to protect an archaeological resource. The site remained under the immediate jurisdiction of the General Land Office until it passed to the newly established United States National Park Service in 1917 making it the fifth park to enter the system.

Frank Pinkley was the first resident custodian assigned to the Casa Grande Ruins in 1901. This move signified the importance of the site. In 1918 President Woodrow Wilson proclaimed Casa Grande a national monument. [8] In 1903 the first shelter was erected over the Casa Grande, a small redwood and corrugated metal roof structure. This overly cramped shelter was replaced in 1932 by a more permanent shelter designed by Frederick Law Olmsted, Jr. and Thomas Vint of the NPS. This structure remains today and is itself now included on the National Register of Historic Places.

The Casa Grande Ruins site benefits from early recognition as a valuable cultural and historic resource. The earliest documentation begun by Father Kino and continued by others in writings, drawings and photographs, provides a useful, nearly continuous record of the site's condition over time. Recently, the University of Pennsylvania, Graduate Department of Historic Preservation in conjunction with CGRNM and the NPS Intermountain Support Office in Santa Fe, Architectural Conservation Projects Program, conducted an in depth 'Wall Condition Assessment Survey' of the Casa Grande. This collection of documentation provides an invaluable resource by which to gauge the integrity and stability of the Casa Grande over

[8] See Clemensen *Centennial History*, p. 35.

an invaluable resource by which to gauge the integrity and stability of the Casa Grande over time and is a base reference point upon which the current conservation programmes for the Casa Grande are developed.

2.1.1 Conservation History
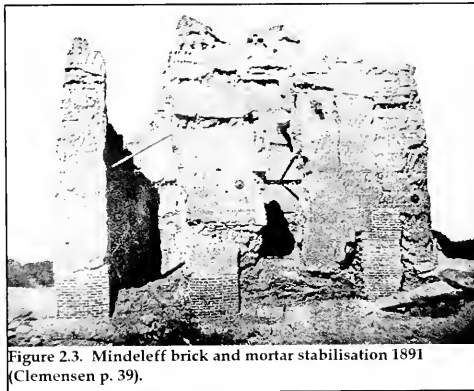
Figure 2.3. Mindeleff brick and mortar stabilisation 1891 (Clemensen p. 39).

In 1890 Cosmos Mindeleff implemented his recommendations for structural stabilisation. He underpinned areas of the exterior south and east walls with brick and cement mortar which was then faced with a cementitious stucco, installed lateral bracing of metal rod and wood from the south wall to the central core of the building, and removed accumulated debris from both the building interior and its immediate surroundings for a ten foot radius. To date, this remains the most significant stabilisation implemented at the Casa Grande.

In 1902 S. J. Holsinger, the local General Land Office agent from Phoenix, AZ visited the Casa Grande to review its condition and meet with custodian Pinkley. Pinkley had changed his mind about the necessity of a roof after the summer rains had eroded more material than he

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[9] Clemensen, *Centennial History*, p. 67.
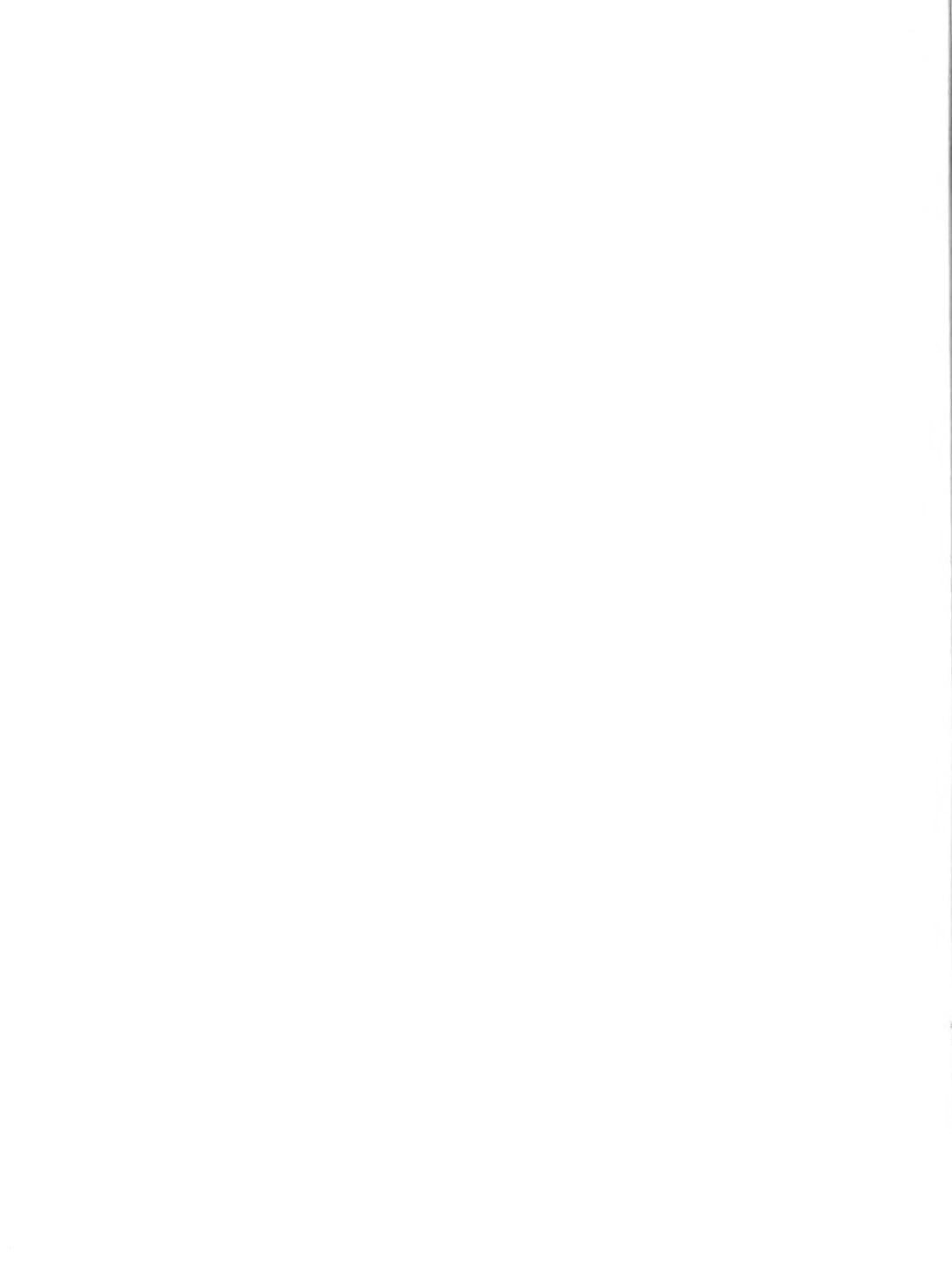
had expected.[10]   While at Casa Grande, Holsinger examined areas of the ruins that Pinkley

had indicated were in need of repair.

Figure 2.4. Cement stucco finish over stabilised areas 1902 (Clemensen p. 41).

> He decided that no more brick or concrete should be used on the ruins as
> Pinkley had proposed, instead any restoration work should be done with as
> much original material as possible. He had Pinkley make a mixture from the
> debris and apply it to seal cracks to see how it worked.   Holsinger
> pronounced the effort a success. Holsinger also indicated a source for future
> repair material. He wrote that Pinkley had recently dug a forty-five-foot-deep
> well.   At the seven-or eight-foot level, he had encountered a stratum of
> cemented gravel which was identical with the original construction material.
> That soil could be used for repairs.[11]

Here is the first explicit conservation philosophy directed towards the maintenance of the site.

Holsinger advocated minimum intervention and emphasized material compatibility.   This

early articulated approach has resulted in an exceptionally high level of integrity and material

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[10] Clemensen, *Centennial History*, p. 50.

[11] Clemensen, *Centennial History*, p. 50.

authenticity at the Casa Grande. Since then, the structure has been subjected to few direct conservation interventions.

The first shelter was erected in 1903, as designed first by Mindeleff, to help protect the structure from further rain damage. The second roof shelter was erected in 1932 as designed by Frederick Law Olmsted Jr. By altering the physical environment immediately surrounding the structure the shelters mediated the forces of weather-based deterioration; an early form of preventative, structurally non-invasive conservation.

Figure 2.5. 1903 shelter, photograph taken 1925 (Clemensen p. 53).
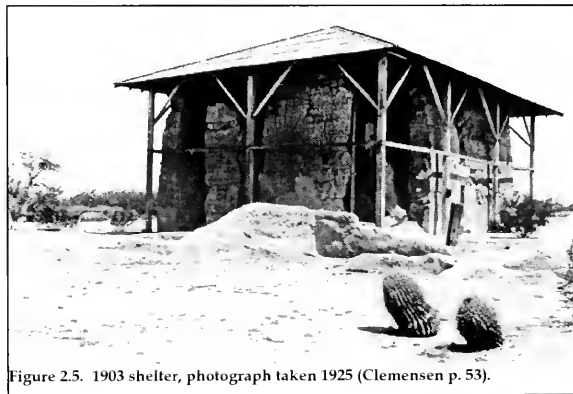
While the shelter has largely removed the problem of direct weathering from rain, it has also visually changed the entire site and its construction has created a microclimate around the ruin that has inadvertently caused some negative impact in its attraction toward birds and other animals.
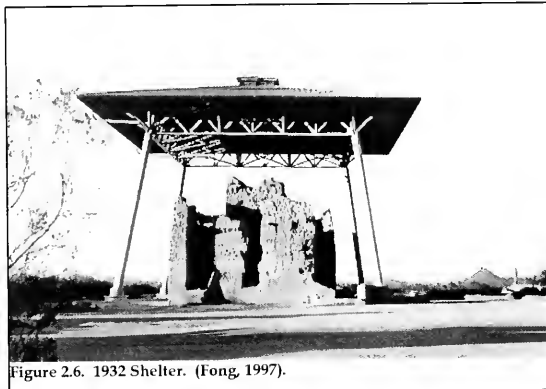
Figure 2.6. 1932 Shelter. (Fong, 1997).

Mindeleff's structural stabilisation and the erection of a shelter are the two most significant physical conservation interventions to the Casa Grande. Other preservation efforts over the years have consisted of closing public access to the interior, minimal soil cement patching and application of water repellents to relatively small areas generally on the south exposure, at the wall tops, or near the fill line of the walls.[12]

2.2   Field Observation/Site Condition

The Casa Grande is exceptional for its integrity, defined as possessing an outstanding degree of original material. The structure retains a high percentage of its interior surface finishes,

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[12] Architectural Conservation Laboratory of the Graduate Program in Historic Preservation, University of Pennsylvania, "Casa Grande Wall Condition Assessment Survey" documents a number of earthen repair patches as 'historic repairs' differentiating them from 'modern' typically soil cement repairs. It is unknown when these historic repairs were administered or by whom but the patch material clearly differs from the surrounding wall fabric.

which are in a predominantly stable condition. According to documentation, little material has been lost since Mindeleff's 1890 initial stabilisation. It has received minimal intervention since its early identification by the United States federal government as a site worthy of recognition and preservation. This federal recognition, along with the early establishment of a conservation methodology of minimal intervention and material compatibility put forth by S.J. Holsinger in 1902, and the erection of protective shelters are the three most significant acts of preservation implemented for the Casa Grande. The original fabrication and construction of the structure, its surrounding climate and the subsequent physico-chemical transformations provide the strong base material to which our material conservation efforts are directed.

Field recordation occurred on three visits to the site, October 1996, December 1996 - January 1997, and August - September 1998. Areas of significant building material loss are not discussed as the focus of this study is the interior surface finish layers, their characterisation, condition assessment, and treatment methods for their stabilisation and reattachment.[13]

The Casa Grande is a massive free standing earthen *caliche* structure. The term caliche identifies a calcium carbonate-based soil. It is believed that the calcareous content contributes to the durability of the Casa Grande through a combination of chemical and physical transformations over time in conjunction with original manufacture and building construction techniques to produce an effect referred to as 'case hardening.' This description indicates an

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[13] For a graphic condition survey of the structure see Architectural Conservation Laboratory of the Graduate Program in Historic Preservation, University of Pennsylvania, "Casa Grande Wall Condition Assessment Survey," 1997-1999.

extremely hard and dense calcium rich zone on the exposed surface of the building material that has developed over time.[14]

The Casa Grande measures approximately 60 feet (north-south) by 40 feet (east-west). Its outer walls rise to an average of 25 feet and the inner walls of the central core rise to an average of 30 feet. The average wall base thickness is 4 feet. This tapers to an average of 2 feet at the wall top. The building is essentially a large multi-storey rectangular space divided into five smaller rectangular spaces. The central core of the structure rises to a height of three stories and is surrounded by adjoining rooms of two stories. These multi-storey rectangular spaces are organized into 'Tiers.' Nothing remains of the wooden beams (vigas) that once supported the earthen floors and divided the vertical spaces. Traces of their existence are still visible as pockets in the walls and wall surface texture differentiation at floor levels. The division of vertical space as would have been defined by floor and roof elements is referred to as 'Room Spaces.'

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[14] For further discussion of this phenomenon see Del Bono, "Characterisation of the Caliche."

Tier: In plan, the Casa Grande is a large rectangular space divided into five smaller rectangles, labeled A, B, C, D, and E. Each smaller rectangle encloses a vertical column of space defined as a tier. Tier D, for example, includes all three of the original floors in that column of space.

A

B   C   D

E

Parapets

Room Space 4

Room Space 3

Room Space 2

Room Space 1

Room Space. The term room space refers to the story-level of the original rooms. Room Space 1 was historically filled with earth, and was not a functional space. The four outer rooms have three room spaces, plus a parapet above the highest roof; the center tier consisted of four room spaces, plus a parapet.

Figure 2.7. CAGR Plan & elevation (Hartzler, 1996).

Beginning with the first descriptions of the Casa Grande, construction methods have been of interest, albeit contradictory. Current research suggests the building was constructed of puddled earth, and built up in horizontal 'courses.' Wilcox and Shenk utilize the term 'course' to describe the distinct horizontal rises of building material distinguished by nearly continuous course boundaries. Course boundaries are through-wall fabrication breaks indicating construction staging, or "the order of construction within a single building episode."[15]

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[15] Wilcox and Shenk, "Architecture of the Great House," p. 69.
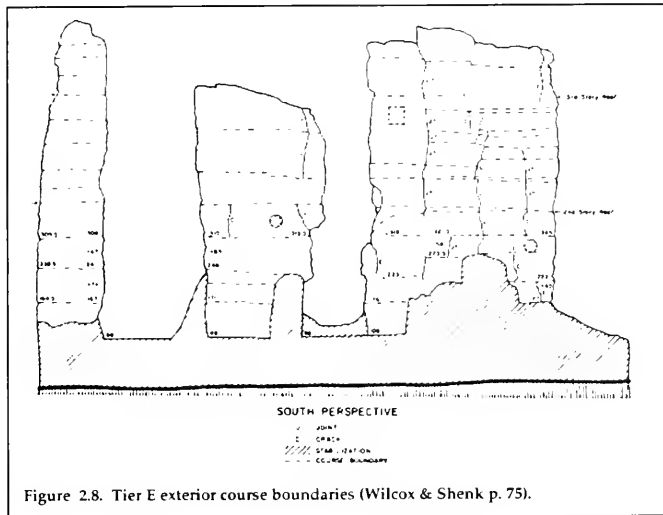
Figure 2.8. Tier E exterior course boundaries (Wilcox & Shenk p. 75).

The process of puddled earth entails the physical breakdown of the caliche material into workable aggregate parts, the addition of water, and kneading into a pliable consistency. "Puddling destroys the crumb structure of a soil and brings soil grains so close together that the movement of air and water is retarded (McGeorge 1937: 128). The volume of capillary pore space is thereby reduced (McGeorge 1937: 128)."[16] In areas of major loss it is sometimes possible to view the substrate construction.[17] What this reveals at times are layers of wall construction consisting of an internal core of "lens-shaped increments."[18]

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[16] In Wilcox and Shenk, "Architecture of the Great House," p. 103.

[17] Major loss as defined by the Architectural Conservation Laboratory, University of Pennsylvania (ACL/UPenn) "Wall Condition Assessment Survey" is at least 10 cm deep and approximately 750 cm³.

[18] Wilcox and Shenk, "Architecture of the Great House," p. 115. For further discussion of construction techniques see also Del Bono "Characterisation of the Caliche."

> The wall courses in both the foundations and the upper walls in the Casa Grande were built up with lens-shaped increments of soil. They often display a curved or "turtle-backed" shape in the upper walls. Their curvature is clearly seen in the wall cross sections and in an especially instructive exposure inside the north wall of tier A. There several facets mark the surface of each lens, and the "dented" appearance of each facet could easily be due to palm impressions resulting from the batches of soil being pressed into place.[19]

Hand impressions inside the wall are clearly visible at numerous locations: Tier C, north wall, room space three, and Tier D, east wall, south facing cross section, room space three. Such imprints contribute to the theory of a puddled earth construction.

Another visible construction phenomenon is the occurrence of interior planar wall layers. These layers appear inconsistently throughout the interior of the Casa Grande. They vary in number, (one to three) and thickness 0.16 - 10.16 cm (1/16 - 4 inches). No pattern of their use has yet been identified although such a determination would be difficult to make without cutting into the wall. At present, the wall layers are visible from exposed broken edges of wall and surface finish (areas of loss). They are clearly a separate application to the interior surfaces of the monolithic wall construction concealing the coursing evident on the exterior by its seams.

What makes both these construction phenomena apparent is their separation or detachment from the surrounding building fabric. The visibility of a soil lens is principally due to loss. Usually, a gap of detachment averaging 3 mm occurs over a visible lens. This indicates a weak interface joint attributable to construction techniques and subsequent shrinkage. Likewise, wall layer construction is observed by the separation of a layer from adjacent building fabric,

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[19] Wilcox and Shenk, "Architecture of the Great House," p. 115.

building fabric, (other layers or the building substrate). The average separation between layers or layer and substrate measures 3 - 5 mm.

The exterior of the Casa Grande reveals exposed caliche substrate. There is no apparent vertical planar layer construction to the exterior facade. The interior spaces of the structure are finished with one to three layers of surface finish comprised of 'intermediary' (leveling) and 'finish' layers. The intermediary layers can number between one and two. As the layers progress outward from the wall substrate towards the surface their aggregate size decreases. This is also reflected in the layer thickness. The final finish layer is always the thinnest and
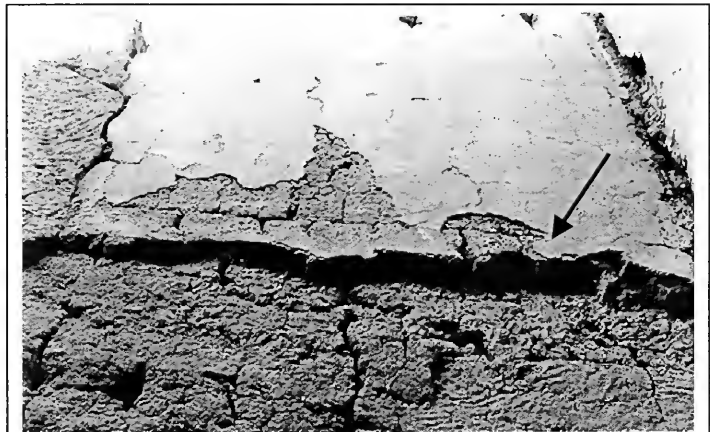
Figure 2.9. Tier B interior, wall finishes layer structure (Fong, 1997).

composed of the finest aggregate. Intermediary layers vary in thickness from 0.6 - 10.16 cm (1/4 - 4 inches). Finish layer thickness range from 0.16 - 0.6 cm (1/16 to 1/4 inch) with an average thickness of 0.16 cm.

17

The finish layer is a largely intact continuous plane of caliche plaster with good internal cohesion. Unlike the smooth, polished surface of the finish layer, the intermediary layer is slightly uneven and rough, useful as a key to the final finish coat. This study focuses on the outermost 1.5 cm of finish (average thickness of samples) with special attention paid to the final 0.16 cm finish layer - as observed in the field - for material characterisation and conservation treatment however, it considers the whole planar construction system as a fundamental factor in understanding the causes of detachment. The final surface finish is a monochromatic pink/reddish wash. The colored wash is not considered to be part of the wall layer construction but applied separately in some cases as several discreet layers, probably over time.

The extant final finish plaster (0.16 cm typ.) is an extremely smooth and even layer across the surface of the interior walls. The term plaster is used here to describe the non-structural, protective finish applied over a

CS = construction seam
S = substrate
Lev = leveling layer
F = finish layer
W = wash

Figure 2.10. Sketch of wall layer construction (2 layers), (Fong, 1999).

Figure 2.11. Sketch of wall layer construction (3 layers), (Fong, 1999).

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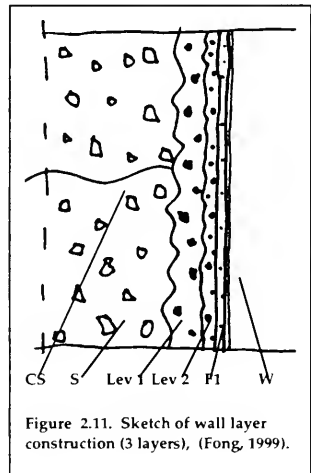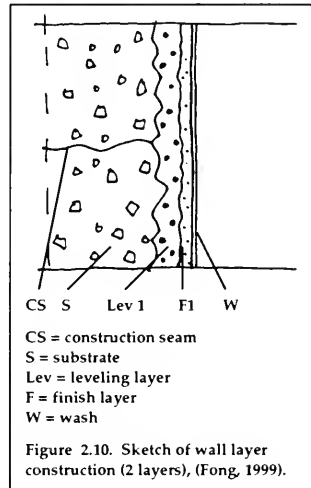
building's substrate core.[20] No application marks are visible. In 1697, three years after Father

Kino's first visit to the site, Captain Juan Mateo Manje noted these finishes,

> There was one great edifice with the principal room in the middle of four
> stories, and the adjoining rooms on its four sides of three stories, with the
> walls five and one-half feet in thickness, of strong mortar and clay, so smooth
> and shining within that they appear like burnished tables, and so polished
> that they shine like the earthware of Puebla.[21]

Some color variation (hereafter referred to as 'darkening') of the finish layer occurs throughout

the Casa Grande. There is no pattern to this variation. Darkening is found on continuous

surface planes absent of any water activity, and along cracks or edges that possibly served as

conduits for water at one time. It is likely that these are two separate conditions. Darkening is

characterized as an integral color change in the surface finish layer. It is not an applied or a

deposited coating such as fire sooting. Areas of darkening around edges and cracks typically

lack their final coat of color wash possibly due to water erosion. In these locations, darkening

may be caused by biological growth. However, darkening that is found within a continuous

surface plane is often covered by the final pinkish/reddish colored wash, the same wash that

covers what have been identified as "historic repairs" or subsequent finishes.[22]

Two types of detachment are evident in the Casa Grande. The first type is an endemic

detachment condition characterised by a fairly even continuous plane of detachment

measuring an average of 3 - 5 mm between two fairly even, continuous adherend surface

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[20] Frank Matero offers the following definition of the term plaster: "...any inorganic binder such as clay, lime,
gypsum, natural or artificial cement used alone or in combination with aggregates that when mixed with a suitable
amount of water forms a plastic mass which when applied to an interior or exterior surface adheres to it and
subsequently sets or hardens, preserving in a rigid state the form or texture imposed during the period of plasticity."
For further discussion of the term, see Frank Matero, "The Conservation of Plasters in Archaeological Sites," *CRM*,
forthcoming.

[21] Clemensen, *Centennial History*, p. 12.

planes. The condition does not appear active and both adherends appear relatively stable. The second type of detachment is less common in the Casa Grande. It is a small scale/flaking condition typically recognized by an actual deformation in the outermost finish plaster layer, frequently accompanied by cracking, and now exaggerated by extrinsic forces such as the pull of gravity, accumulation of debris or animal activity. This type of detachment is the more critical of the two (termed active) as the now deformed fragment hangs in a precarious state. (The possible mechanisms of detachment and deterioration are discussed in Chapter 3.)

Tier C is distinguished from the four other tiers by possessing the highest integrity of surface finish. Integrity is measured by the amount and condition of extant finish. The outstanding condition of Tier C is attributable to its protected location at the core of the building.

Tiers A, B, D, and E all display a fine particulate dust coating on their surface. It is unclear what the origins of this fine matter are. Probably it is a result of wind blown deposits or decomposition of the wash layer as it loses its internal cohesion, or a combination of both. Tier C is sheltered from extrinsic environmental forces by its central location. This not only shields it from windblown deposits but from the erosive forces that deteriorate the cohesive strength of the finish.

Tier E suffers less from detachment than from surface erosion. This is the southernmost tier and is subjected to the greatest force of the weather that typically approaches from the

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[22] See ACL/UPenn, "Wall Condition Assessment Survey."

southwest. Much of the pink/reddish color wash of Tier E has eroded off of the finish plaster layer.

Tier B exhibits the greatest amount of detachment of all five tiers. The reasons for this are unclear. Tiers A and D are fairly comparable in the condition and characterisation of the surface finishes. The surface finishes in these tiers are largely intact except for areas of major loss, with occasional areas of detachment. Tiers B and D retain evidence of active liquid water ingress. It is uncertain when this occurred although it most likely predates the present shelter.

Evidence of animal activity is present throughout the Casa Grande in the form of spider webs, (intricate networks of spider webs are frequently found in and around cracks and holes), guano and traces of rodent tunnels previous to the debris/fill removal. These tunnels are visible on the surfaces of a few of the walls, just below the fill lines. Animal activity poses a threat to areas of detachment. Burrowing or nesting in detachment gaps exacerbates the active or potential instability of the detached plaster.

2.3   Material Characterisation

The purpose of material characterisation is manifold. It is one critical step in the documentation and investigation of a site or structure. Materials are characterised physically and chemically. Material characterisation informs a comprehension of the historic structure as it yields the potential to reveal building sequence, design, fabrication, methods of application, and occasionally material provenience when matched with archaeological research.

Knowledge of material characteristics is essential for the development of an appropriate treatment programme. In this investigation, material characterisation was performed primarily for documentation, treatment, and architectural comprehension purposes. Of the latter, the small size of the samples removed from the site greatly limited the information that could be gleaned but used in conjunction with past and possible future studies it can provide valuable corroborating information. Sample size and availability also limited the methods of material characterisation investigation. All characterisation was performed via visual bulk and microscopic analysis.

The laboratory material characterisation process was divided into three steps; bulk analysis and micromorphological analysis - cross section and thin section.

2.3.1   Sampling

The goal of the sampling programme was to evaluate and determine the finish plaster layer construction, microfabric structure, and an assessment of similarities and differences amongst room spaces and tiers. A sampling strategy was carefully planned that would accomplish these goals with the minimum amount of extraction and intrusion. Tier, room space, orientation (north, south, east, west), elevation (wall top, middle, base), and architectural location (wall surface, inside doorway, door plug, solstice hole) were amongst the considerations in the design of the sampling programme. Interior samples were selected to represent the various conditions found in the Casa Grande, both typical and extreme. These included what was perceived to be the most intact surface - that with a strong cohesive wash; finish layers of apparent varying thickness; darkened and non-darkened areas, and areas of historic repair. The average size of each sample was approximately $2.5 \times 1.5 \times 2$ cm or 7.5 cm^3.

Forty samples of the finished interior walls were taken in the fall and winter of 1996. Samples were taken by the author, Robert Hartzler (NPS) and Frank Matero (UPenn). A large portion of the fallen exterior wall fragment was also packed and sent to the Architectural Conservation Laboratory (ACL) at the University of Pennsylvania for characterisation and testing.[23] The exterior wall fragment was used exclusively to fabricate assemblies for treatment testing of adhesive grouts. (See Appendix 10.1 Sample Map)

Selection was always based upon the potential or ability to yield useful information. What is deemed useful information is entirely dependent upon the questions posed. Here, the critical questions identified were:

- What is the physical, micromorphological structure of the fabric?

- What is the mineralogical composition of the material?

- Is there a distinct layer structure visible within the sample?

- What are the characteristics of the perceived 'darkened zone?'

  - Is it a distinct layer?

- Is there a difference in material and micromorphological structure of the finish layer amongst room spaces and/or tiers?

- Are there multiple layers of color wash?

- Is there a distinct difference in material and structure between the finish layer and the building material?

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[23] See Del Bono, "Characterisation of the Caliche."

2.3.2   Bulk Analysis

All samples were bulk analyzed and examined visually with a stereo binocular microscope (Nikon SMZ-U) under quartz halogen reflected light illumination at a total magnification of 7.5x unless otherwise specified. Bulk analysis produced a gross physical characterisation of the samples and allowed sample comparisons and selection for subsequent analysis. The questions posed were;

- What is the layer structure?
- Is there a wash layer?
  - What is its condition?
- Is there a darkened zone?
- What is the aggregate profile (shape, size and general distribution)?
- What is the matrix profile?
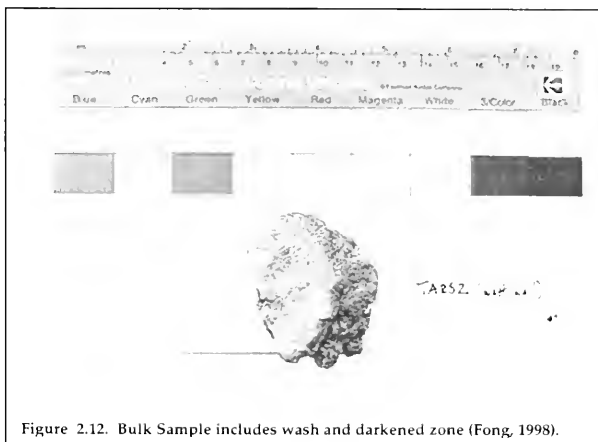
Figure 2.12. Bulk Sample includes wash and darkened zone (Fong, 1998).

Finish samples ranged in thickness from 4 mm – 4 cm with an average thickness of 1.5 cm. The colored surface wash occurs as a distinct layer, very thin, ranging in thickness from 0.02 – 0.376 mm.[24] Apart from the applied colored wash layer, no distinct internal stratification was evident within an individual sample. However, given that the average sample thickness is 1.5 cm this clearly represents only the finish layer and possibly a portion of the outer leveling layer of the planar wall layer construction (see Figures 2.10 and 2.11). Therefore, what is observed in the samples does not represent the vertical planar wall construction in its entirety but rather a limited portion. The small sample size was a result of the sampling programme that stressed minimal impact (extraction/intrusion). While thicker samples would have been helpful in the assessment of layer construction, they were not deemed essential for material characterisation in the development of a treatment grout. For the purposes of description, the term 'zone' is used. Zone indicates a subtle differentiation from one area of the sample to another, regarded in strata from the exterior surface inward. There were no clear color or microfabric distinctions from one zone to the next yet differences were perceptible from the outer most to the inner most zones. As observed in the field, some samples contained a darkened charcoal-grey zone just beneath the wash layer yet even this visually apparent zone was indistinguishable from the immediately adjacent finish material (depth into the sample). It was distinct in color from the bulk of the sample but did not exist as a discrete layer. The matrix and aggregate profiles amongst the samples were similar except for the presence of a darkened zone or the inclusion of an occasional large caliche nodule.[25] Aggregates are heterogeneous in color, size, and mineralogical composition, primarily subangular and

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[24] These measurements were calculated from photomicrographs taken at 100x and compared against a relative micrometer scale.
[25] Caliche nodules are large carbonate inclusions within the building fabric.

subround in shape, and poorly sorted. The fine matrix was typically a brown to brownish grey color. Twenty-four samples were selected from the original 40 for cross section analysis. These twenty-four samples were embedded in an acrylic polyester resin (Bioplast™), cut into cross sections with a Beuhler Isomet[5] and polished for further microscopical analysis. (See Table 2.1 Bulk Analysis Sample Characterisation.)

2.3.3   Cross Section Analysis

Cross section analysis was performed using a Nikon AFXIIA with quartz halogen fiber optic reflected light illumination at a total magnification of 40x unless otherwise specified. An initial cross section test sample revealed that the samples would first have to be consolidated in order to withstand the friction and constant wetting during the cross section cutting process. This initial sample crumbled and disintegrated during the process. All twenty-four samples were consolidated with a 5% solution of Acryloid B72 in acetone w/w (i.e. 5g B72 + 95g acetone). The samples were set in a flat dish on a bed of glass beads. The B72 solution rose to just beneath the top of the glass beads. The samples were allowed to absorb the solution through capillary rise over a period of 36 hours. Samples were turned and the consolidant solution replenished every 12 hours. Once dry, the twenty-four samples were embedded in Bioplast and cut on a Beuhler Isomet[5] low speed saw. The first cut revealed that the consolidant had not fully penetrated into the center of the samples. Each sample was therefore cut open and consolidant solution dropped onto the newly exposed surface to the point of complete saturation. Samples were again cut to form cross sections and polished with stoddard solvent on 400 and 600 grit polishing strips and Beuhler 'microcloth' polishing cloth

disks. The shallow penetration of the consolidant indicated a significant density and low porosity of the material.[26]

The questions posed during cross section analysis were the same as those posed during the bulk analysis phase however the investigation was performed at a more refined level. Cross section analysis revealed,

- the finish is a dense material with low porosity

- the color wash is a distinct, applied layer

- the darkened zone is not a distinct layer and typically measures 2 mm in depth

- the aggregate profile is heterogeneous in color, size, and mineralogical composition, primarily subangular and subround in shape, and poorly sorted (thus confirming bulk analysis profile)

- the matrix color varies from tan, brown to grey without clear distinctions

- the absence of a distinct layer structure within the fabric promotes the usage of 'zones' to describe the gradual change in color from the outermost finish surface to the sample interior.

- channel voids occupy 5% of the sample[27]

Figure 2.13. Channel voids (Courty p. 72).

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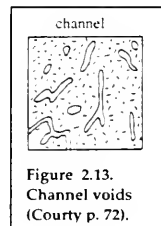
[26] Whether the low porosity is attributable to the low percentage of pores or the dimension of those pores was not yet clear. Subsequent cross section analysis revealed a low percentage of pores, approximately 5%.

[27] P. Bullock et al., *Handbook for Soil Thin Section Description*, (England: Waine Research Publications, 1985), Defines channel voids as "Elongated, cylindrical or arched, regular conformation, usually smoothed walls; uniform cross section over much of length," p. 43.

27

BULK ANALYSIS SAMPLE CHARACTERISATION

Sample no. *	Tier	Room space	Orientation	Architectural function & location	Layer thickness**	Strata***
5	A	2	north wall	top of wall near corner	6-12 mm	W=y, D=y
14.2	B	2	south wall	door plug face	no notes	W=y, D=n
12	B	2	west wall	top of wall near vega line	possib. 12-25 mm	W=y, D=y
(10.96) 6	C	2	east wall	wall	approx. 1.5 mm	W=y, D=n
(10.96) 7	C	2	east wall	wall near base	approx. 1.5 mm	W=y, D=n
19.1	B	3	south wall	door plug face	no notes	W=y, D=p, O=Z2 p lt. grey
20.1	C	3	south wall	wall	approx. 3 mm	W=y, D=n, O=Z2 p lt. grey
1	A	2	west wall	wall at corner	approx. 12.7 mm	W=y, D=y
(10.96) 16	B	2	south wall	inside door frame	no notes	W=y, D=n
8	B	2	north wall	wall vega rest	no notes	W=n, D=y
(10.96) 1	D	2	north wall	partition wall at corner	no notes	W=y, D=p
19	D	2	west wall	wall near corner	6.35-25.4 mm	W=y, D=n
17	E	2	north wall	wall	no notes	W=n, D=n
18	E	2	south wall	wall	10 mm	W=y, D=p
6	A	3	west wall	ceiling of solstice hole	no notes	W=y, D=p
18.1	B	3	south wall	inside door frame	no notes	W=y, D=n
11	B	3	west wall	inside door frame lintel level	approx. 10 mm	W=?, D=n
9	B	3	west wall	wall	76.2 mm	W=y, D=?
17.2	B	3	south wall	wall	no notes	W=y, D=y
10	C	3	east wall	wall	6.35 mm	W=y, D=n
15	E	3	south wall	wall near base	76.2 mm	W=y, D=p, O=Z2 p lt. grey
14	E	3	south wall	top of wall near vega line	19.05-25.4 mm	W=n, D=?
(10.96) 11	C	4	east wall	wall near base	6.35-12.7 mm	W=y, D=?
(10.96) 12	C	4	east wall	wall near base	6.35-12.7 mm	W=y, D=y

*These first seven samples were selected for thin section analysis

**These (usually estimated) measurements are taken from field notes and refer to the presence of a finish layer.

***W = wash, D = darkened zone, y = yes, n = no, O = other, Z = zone, p = possible

28

Table 2.1

Colored wash, slightly blistered

Darkened zone, dark grey

Small isotropic particles, possibly carbon.

Large caliche nodule itself, heterogeneous in composition.

Channel void

It is doubtful that this represents a layer interface as it does not clearly travel across the entire sample. The photomontage does not represent the entire width of the cross-sectioned sample. Many samples suffered trauma during the cross-section cutting process. Some of the visible microfabric interior cracks could be due to physical trauma experienced during the cutting process.

Channel void

Figure 2.14. Sample 12 Cross section, 10x magnification, quartz halogen reflected light illumination (Fong, 1998).

As viewed in cross section, the micromorphological structure of the finish layer is comprised of heterogeneous aggregates of various shapes and sizes, unevenly distributed throughout the fabric. The wash is clearly a

very thin applied layer. The darkened zone is not a specific layer but rather an intrinsic coloration within the finish layer.

From the twenty-four samples surveyed in cross section, seven were selected for thin section analysis (TARS2 no. 5, TBRS2 no. 14.2, TBRS2 no. 12, TCRS2 (10.96) no. 6, TCRS2 (10.96) no. 7, TBRS3 no. 19.1, and TCRS3 (10.96) no. 20.1). Bulk and cross section analysis revealed more similarities than differences amongst the samples. Micromorphological characteristics - color wash, darkened zone, calcite nodules - as well as architectural context served as the guidelines for final thin section selection.

2.3.4   Thin Section Analysis

The selected seven samples were sent to Spectrum Petrographics, Inc. for thin section production. All samples were vacuum impregnated with clear epoxy, cut to standard thickness (30μm); half of each sample was stained for calcite with alazarin red, and affixed with permanent glass coverslips. Microscopic analysis was conducted with a Zeiss Axiophot under plane polarized and cross polarized light at 25x, 40x and 100x magnification at the University of Pennsylvania, Department of Earth and Environmental Science. Photomicrographs were taken at 25x and 100x.

Thin section analysis permits a more detailed investigation into the qualitative and quantitative characteristics of the microfabric of a material. Particle size analysis and mineralogical identification were performed during this phase of characterisation.

Observations made during the different phases of material characterisation can serve to corroborate and/or challenge hypotheses and assumptions.

Wash layer distinct however, multiple layers not visible.

Poorly sorted heterogeneous aggregates.

Small isotropic particles, possibly carbon.
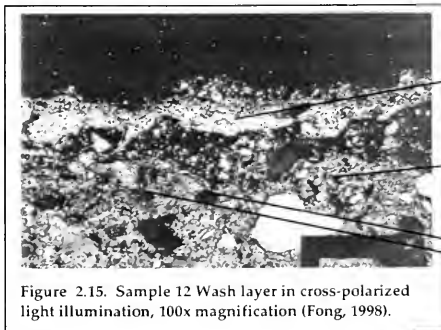
Figure 2.15. Sample 12 Wash layer in cross-polarized light illumination, 100x magnification (Fong, 1998).

What appeared as possible multiple layers of color wash in bulk and cross section analysis were not clearly discernible in thin section under cross-polarized light however, plane polarized light revealed details that suggested multiple layers were present. The darkened zone visible in bulk and cross section was not perceptible in thin section; this is possibly due to the thin section sample size, which was often smaller than the cross sections and always smaller than the bulk samples. The mineralogical and physical composition of the surface finish appeared nearly identical to that of the caliche building fabric except in particle scale.

Applied wash collected in divot of surface. There is some suggestion of stratification of wash layers
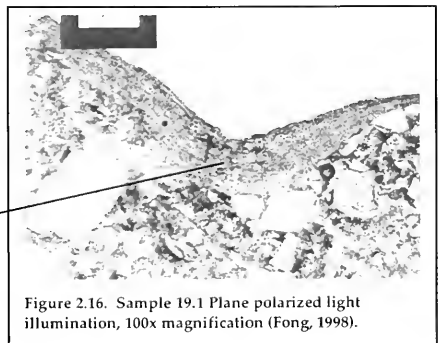
Figure 2.16. Sample 19.1 Plane polarized light illumination, 100x magnification (Fong, 1998).

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The non-destructive method of point counting was selected to conduct particle size analysis (PSA) of the samples. Standard PSA tests performed through physical gravimetric analysis were not possible due to limited sample material and sample size. The point counting method relies on photomicrographs taken at 100x against a relative micrometer scale. No less than 55 particles were counted per sample. The average thin section surface area is 1.22 cm.

Criterion for particle selection was the range of sizes as defined by ASTM convention.[28]

Gravel	75 mm	-	4.75 mm
Coarse sand	4.75 mm	-	2.00 mm
Medium sand	2.00 mm	-	0.425 mm
Fine sand	0.425 mm	-	0.075 mm
Clay & Silt (Fines)		<	0.075 mm

The length of the long axis determined particle size regardless of orientation. The rationale for this determination is based on physical (gravimetric) methods of particle size analysis. These methods involve the disaggregation and sieving of the material through a standard set of varying screen sized sieves. In this case it is the long axis of a particle that is the greater determinant in its passage from one screen to the next.

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[28] "D422, Standard Test Method for Particle-Size Analysis of Soils," (Philadelphia: ASTM, 1993); and "D2487-85 Standard Test Method for Classification of Soils for Engineering Purposes," (Philadelphia: ASTM, 1985). This standard states that clay is a soil passing a No. 200 (0.075 mm) U.S. standard sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents and that exhibits considerable strength when air dry. A silt is defined as a soil passing a No. 200 (0.075 mm) U.S. standard sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry.

Particle Size Range Percent Fraction*

Sample T= Tier RS = Room space	Coarse Sand 4.75-2.00 mm	Medium Sand 2.00-0.425 mm	Fine Sand 0.425-0.075 mm	Fines[†] < 0.075 mm
TARS2 no. 5		10%	57%	33%
TBRS2 no. 14 2	2%	11%	55%	32%
TBRS2 no. 12		16%	56%	28%
TCRS2 (10.96) no. 6		16%	46%	38%
TCRS2 (10.96) no. 7'		13%	47%	40%
TBRS3 no. 19.1		11%	54%	35%
TCRS3 (10.96) no. 20.1		13%	55%	32%

* All percentages are rounded to the nearest whole number.
[†] This fraction is probably much higher than recorded as it should technically be considered matrix.
' Size of thin section should be noted when considering this %. The surface area of this sample (no. 7) is approximately 0.4 sq. mm compared to an average of 1.28 sq. cm.

Table 2.2

Particle size range percent fraction is calculated by the following equation,

% = (no. of particles in size range / total no. of particles measured) x 100

Quartz

Strained feldspar

Fine grains of quartz in a matrix of fine-grained material.

Calcite nodule stained red.

Crypto-crystalline calcitic matrix

Figure 2.17. Sample 12 Cross-polarized light 100x magnification (Fong, 1998).
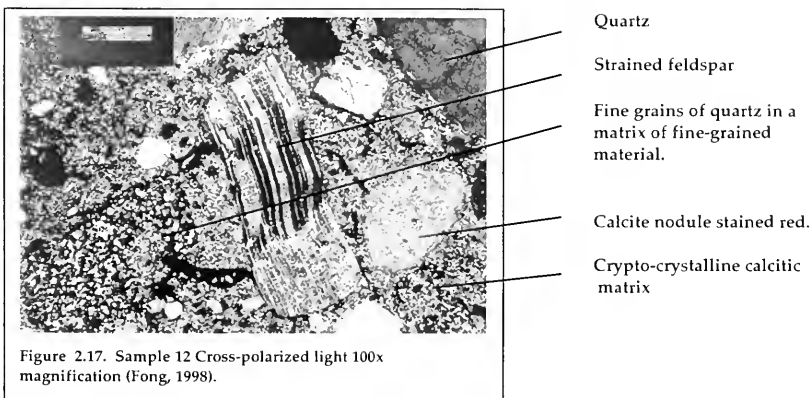
The primary aggregates of all samples are quartz, feldspar and calcite nodules. Alazarin red staining reveals a low to moderate presence of calcite in the fabric matrix. The calcite is otherwise concentrated in caliche nodules found as aggregates within the matrix (see Figure 2.15 and 2.16). The wash layer is not high in calcite as it did not retain the stain. It is

composed of extremely fine particles, possibly composed of mineralogical clays (see Figures 2.13 and 2.14).

Dark red staining of caliche nodule indicates high calcite content unlike the low-medium staining of surrounding matrix.

Channel voids

Figure 2.18. Sample 12 Cross-polarized light illumination, 25x magnification, 2/3 of sample in photo is stained with alazarin red for calcite. (Fong, 1998).
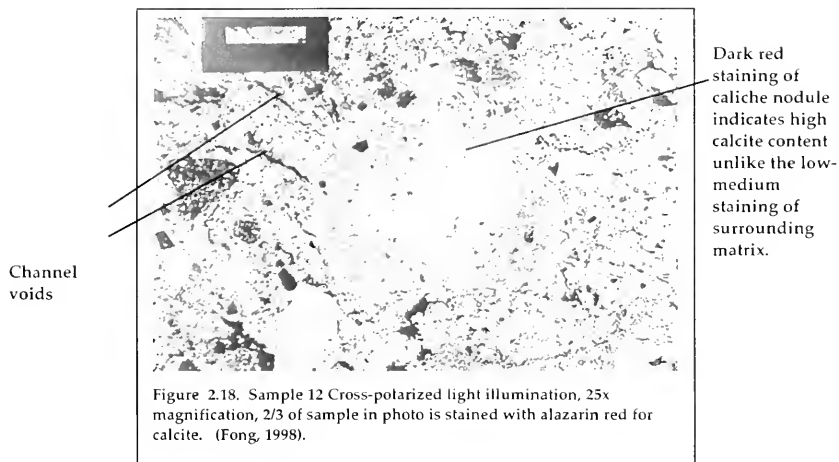
Micromorphological analysis produced the following characterisation.[29]

- The finish is a fairly dense caliche material containing approximately 5% voids in primarily channel form.

- The material is constituted of approximately 28% aggregate (fine and medium sands fraction).[30]

- The aggregate fraction is comprised of an average of 53% fine sand and 13% medium sand.

- The primary aggregates are quartz, feldspar and calcite nodules.

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[29] This characterisation represents a typical sample. The following characterisation percentages are therefore averages of the samples analysed.

[30] This percentage was determined by comparing photomicrographs of samples taken at 25x magnification and viewing thin sections at 40x magnification against a standard chart "to estimate the abundance of objects seen in the Microscopic field." See Marie Agnes Courty et al., *Soils and Micromorphology in Archaeology*, (Cambridge: Cambridge University Press, 1989), p. 69

- The aggregates are poorly sorted throughout the matrix.

- Calcite is found within the matrix in a low to moderate amount.

- The majority of calcite is located within calcite nodules found as aggregates lodged within the matrix, but also exists as crypto-crystalline calcite in the matrix.

- The color wash is not calcitic, is very fine, and is most likely comprised of mineralogical clays.

- There is no distinct layer structure within the outermost 1.5 cm.

- At this point, the darkened zone is visible as a chromatic change only in bulk and cross section analysis. Two possible causes of this integral and subtle color change gradation of the finish layer are physico-chemical transformation as induced by high heat (fire) or fabrication methods.

- There does not appear to be a difference of material amongst tiers nor room spaces.

- The mineralogical and physical composition of the surface finish appears nearly identical to that of the caliche building fabric except in particle scale.[31].

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[31] See Wilcox and Shenk, "Architecture of the Great House," p. 93-97 for Edwin R. Littmann's analysis of Casa Grande soil samples. Littmann's 1967 study reached the same conclusion regarding the physico-chemical similarities between the finish and building material.

3.0 TREATMENT REQUIREMENTS

3.1 Identification of the Conservation Problem

Two types of surface finish detachment conditions are in evidence at the Casa Grande herein identified as Type A and Type B. Type A (large scale) is the more pervasive detachment condition of the two and found throughout the structure. It is characterised by a fairly continuous plane of detachment measuring an average of 3 - 5 mm between two fairly even,

continuous adherend surface planes (intralayer or finish layer to substrate). Layer thickness ranges from 0.16 - 0.6 cm (1/16 - 1/4 inch) for finish to 0.6 - 10 cm (1/16 - 4 inches) for intermediary (see Figures 3.1 and 3.2). The condition appears to be relatively stable (inactive). Type B (small

Figure 3.1. Tier E, Type A detachment (Fong, 1998).

scale/flaking) is the less common detachment condition and is recognized by an actual deformation in the outermost finish layer. It is frequently found in conjunction with cracking and is easily exacerbated by animal activity and debris accumulation. This condition primarily occurs on a small, localised scale (see Figures 3.3 and 3.4). This type of detachment is the more critical of the two as the now deformed fragment hangs in an unstable, precarious state. This instability characterises this condition as active.
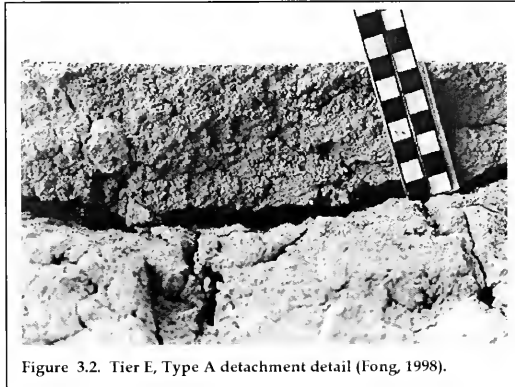
Figure 3.2. Tier E, Type A detachment detail (Fong, 1998).

Detachment is visible from broken exposed edges of fragments and walls (areas of loss). It is therefore impossible to accurately determine the extent (breadth) of the detachment gap associated with Type A (largely a blind condition). The extent of detachment visible in Type B is discernible through associated surface conditions such as mapcracking, deformation and surrounding areas of high loss.
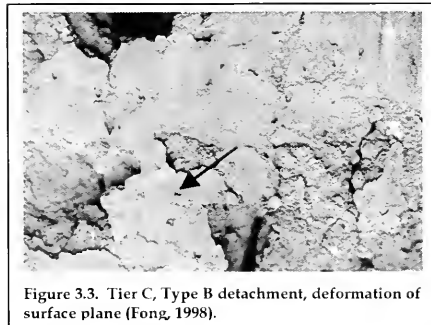
Figure 3.3. Tier C, Type B detachment, deformation of surface plane (Fong, 1998).
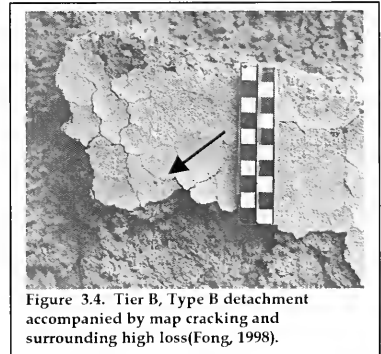
Figure 3.4. Tier B, Type B detachment accompanied by map cracking and surrounding high loss(Fong, 1998).

In the development of a treatment programme both detachment conditions were considered however, a decision was made to focus on the variables of Type A due to its prevalence and

unusual nature. Treatment methods have already been developed and successfully implemented for the small-scale detachment of earthen plasters (Type B). If the gap of detachment and the thickness of the detached layer are small enough, very often the most successful method of reattachment is rehydration of the earthen material. Water is the natural plasticizer of clay. Treatment is effectively administered by misting or injection of a mixture of ethanol and water and gently pressing the detached area back into alignment with the plaster plane. In arid environments, small earthen fragments of narrow detachment gaps have also been reattached using a low concentration of gelatine (1.25 % w/v). An obvious concern associated with this method is biosusceptibility. Both of these methods, water and ethanol, and gelatine have been successfully used on thin earthen finishes at Mesa Verde National Park.[1] If the gap of detachment is too great or the finish layer too thick to be pressed back into alignment, then an adhesive with gap filling properties becomes necessary. This latter case presents the treatment requirements of Type A. Therefore, if Type B cannot be treated with known methods then the results of an investigation for Type A condition could prove useful.

Type A is unusual in the expanse and regularity of the detachment gap and the regularity and thickness of the adherends. The seemingly large, continuous expanses of detachment and/or the thickness of the detached layer(s) defy typical rehydration reattachment methods of realignment. The nature of the detachment raises a number of conservation treatment issues. (The possible causes of the condition are discussed below in 3.2 Mechanisms of Deterioration and Detachment.)

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[1] See Architectural Conservation Laboratory (ACL), University of Pennsylvania, "Condition Assessment of Architectural Finishes, Mug House, Mesa Verde National Park, Mesa Verde, Colorado," 1995-1996, documentation

- What causes this condition and when did it occur?

- Is the condition active?

- Is the condition stable?

- Does the situation require stabilisation and/or reattachment?

- Is it desirable to fill the gap with a continuous film or is it preferable to "spot weld"?

- What are the points of access and how will treatment be administered/applied?

3.2 Mechanisms of Deterioration and Detachment

Detachment is typically observed at exposed broken wall or fragment edges (areas of loss); otherwise it is blind. The causes for loss and the causes of detachment are not necessarily one and the same. The majority of visible surface finish loss probably predates 20th century shelters for the causes of this condition are almost certainly related to abandonment and lack of maintenance resulting in unmitigated exposure to wind and rain, extraction of wood members (vigas and lintels), floor/roof collapse, removal of fill and subsequent climatic change resulting in basal erosion, animal activity, and vandalism. There has been occasional but minimal loss of material since the erection of the shelters, however some areas remain vulnerable due to their precarious state created by earlier damage. Type B detachment is almost always directly related to these factors.

drawings. ACL, University of Pennsylvania.

The highly regular and even nature of Type A detachment suggests that it is a result of original construction techniques (see Figures 3.1 and 3.2). The perceived planar detachment may in fact be a separation induced by shrinkage rather than post construction trauma such as that caused by seismic vibrations. The same phenomenon of shrinkage is what permits us to view the lens and layer construction of the wall substrate and is a contributing factor to the subsequent loss of fabric. The gap of detachment is visible as a result of loss. Loss has occurred at that location in part because of the lack of support evidenced by the gap. These are mutually descriptive and dependent conditions. What might have previously passed undetected or otherwise noted as a blind void/detachment now becomes visible and susceptible to extrinsic mechanisms of decay. If in fact, as it appears to be, the detachment is a result of construction technique then it is an original condition, clearly inactive, and therefore stable. However, while the condition may be relatively stable, it still presents an extensive plane of discontinuity within the walls. With loss, exposed broken edges become points of vulnerability for further loss induced by such agents as water penetration, debris accumulation, insect and animal activity and physical/mechanical impact caused by visitor intervention.

Shrinkage between two layers of like material occurs when the moisture contents of those two layers are too dissimilar. The imbalance of moisture levels induces rapid evaporation caused by suction of the moisture out of that material with the higher moisture content by and into the adjacent less hydrated material. Rapid evaporation inhibits the bonding process between the two surfaces and frequently results in shrinkage (contraction and consequent reduction of interfacial contact) and cracking. Clearly, the intra layer separation is not continuous

otherwise wholesale failure would result. There is no evidence of mechanical bonding between layers although surface roughness may have been intentional in order to induce keying. Type B failure is probably due to a finer texture and less well graded aggregate component in the outer finish layer (compared to the adjacent layer) combined with the stresses caused by wetting and drying after abandonment and exposure.

Type B detachment is clearly a subsequent aberration that has increased in extent and severity over time. Its current active and unstable condition is identifiable as failure. By contrast, Type A detachment may have existed since the point of original construction, in which case it is not an aberration. Its condition is inactive, has not increased, and therefore is not necessarily definable as failure by the same process of reasoning. However, the pervasive nature of Type A detachment does pose potential of extensive failure (loss). Extrinsic factors such as seismic tremors could cause widespread failure for this currently inactive condition. As previously mentioned, Type B detachment should immediately be addressed and treated. A policy of treatment for Type A will require consideration of many factors specific to the site. For Casa Grande these include:

- The statistical possibility of seismic tremors
- The high integrity and original material authenticity of the Casa Grande
- The opportunity to monitor and assess trial treatments if administered.

All factors must be balanced and their relative benefits weighed against the potential disadvantages of intervention to such a stable condition when motivated by a statistical unknown (likelihood of seismic tremors). Preventative maintenance is indisputably valuable,

the points of and occasions for interventions must be wisely and discretely selected and limited to those of maximum affect with minimum impact upon the site and structure.

3.3 Treatment Requirements

The conservation problem is the *in situ* stabilisation and reattachment of the finish layer(s) in the Casa Grande. The variables are caliche fabric (for both adherends), an average detachment gap of 3 mm and limited points of access. The majority of exposed gaps are accessible from the bottom edge upwards. Upward facing edges are typically covered over and/or filled in with debris carried downwards by gravitational force. What is desired is an injectible adhesive with gap-filling properties (grout) that is chemically, physically and mechanically compatible with the original fabric. Philosophical and theoretical criteria of intervention advocate minimum intervention towards the maximum retention of original fabric.

The ideal grout fulfills the following criteria,

- Compatibility
- Stability
- Adhesive bond strength
- Cohesive strength
- Minimal shrinkage
- Water vapor permeability
- Low biosusceptibility

- Minimal weight
- Injectibility
- Flow
- Thixotropy
- Working time
- Ease of use and disposal
- Retreatability

Compatibility

The selected grout must be chemically, physically and mechanically compatible with the surrounding original fabric. It should not drastically alter the inherent properties of the adherends nor should it pose too foreign a barrier within the existing system. Chemical compatibility implies a sympathetic bond of materials on the chemical level, both initially and over time. The aim is to work with the nature of the original material, not to change it unless it is inherently compromised. Ideally, the chemical composition of the treatment material should share the fundamental chemical characteristics of the original fabric. Given the high $CaCO_3$ content of the adherends, a lime-based grout would be appropriate; however the detachment is too narrow to use lime based grouts which require a minimal aggregate size still too large for injection into these narrow voids. Physical compatibility indicates a sympathetic relationship between the original fabric and the treatment material such as porosity and permeability. Mechanical compatibility refers to functional operations in a systemic or compositional form such as bond strength. Does the treatment perform its mechanical function in a manner that is effective and beneficial to the original system? The treatment must perform its desired purpose without causing additional stress or undue chemical and/or physical change to the existing fabric and system.

Stability

The treatment material should be stable over time in an uncontrolled exterior environment (i.e. one subjected to temperature and humidity fluctuations). It should perform its function

in the state in which it was designed. Adhesive bond failure, creep, flex, and/or change in pH are examples of instability and indicate a degradation and weakening of the system.

Adhesive Bond Strength

Proper adhesive bond strength provides adhesion and associative support but is never stronger than the original fabric itself. The point of failure under load for any reattachment adhesive conservation treatment should be at the interface between adhesive and original fabric or preferably within the treatment material itself. These points of failure are designed to prevent further loss or harm to original fabric. The system is only as strong as its weakest member.

Cohesive Strength

Cohesive strength describes the internal strength of the treatment material. The treatment material must have good cohesive strength before it can offer adhesive strength. Good cohesive strength is a result of even particle distribution and a strong network of physical and chemical bonding.

Minimal Shrinkage

The treatment material should display minimal shrinkage. It must retain its form in order to maintain interfacial contact and adhesive bonding with the surrounding fabric as well as cohesive strength. Too much shrinkage is a failure of the system.

Water Vapor Permeability

Water vapor permeability is the ability of a material to permit the passage of water vapor. This is an essential property. Any impermeable barrier will have deleterious effects, causing greater damage than benefit. Trapped liquid water or condensation at the interface can lead to softening or collapse/failure of the earthen material, formation of salts, biological growth, and damage due to freeze/thaw. Any or all of these effects can cause further detachment or deterioration amounting to loss.

Biosusceptibility

The grout should have zero to low biosusceptibility. There is no evidence of microflora on site given the low moisture levels and shelter protection.

Minimal Weight

The grout, while not structural is intended to provide a measure of support by providing a backing for the detached layer. It must have sufficient cohesive strength in order to offer support however, it must be light in weight to avoid contributing undesired additional weight to the already vulnerable system.

Creep

The grout should not experience creep. Creep is "the dimensional change with time of a material under load, following the initial instantaneous elastic or rapid deformation."[1] While

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[1] "D907, Standard Terminology of Adhesives," (Philadelphia ASTM, 1990), p. 35.

it is virtually impossible for plastic materials to be absolutely free of creep, the degree to which they do exhibit this characteristic should be negligible. A degree of flexibility is desirable yet the grout should not 'sag' from its original location. This issue is initially addressed in the design of a thixotropic grout. Creep primarily addresses dimensional change as a result of load however dimensional change can also be induced by a rise or drop in temperature. An expansion of the grout will exert stress upon the adherends resulting in eventual cracking, (failure) and future loss; contraction of form is addressed in 'shrinkage.'

Injectibility

Injectibility, flow and thixotropy are characteristics related to application. Limited access to detachment gaps requires a highly injectible grout. The 3 mm size of the typical gap requires injection through a hypodermic syringe and gauge 16 needle (1.65 mm outside diameter). Any adhesive formulation must be fluid enough to pass through these tools.

Flow

The grout must have good flow. Flow is the movement ease and ability of a liquid or semi-liquid material. This is directly related to the viscosity and surface tension of the adhesive formula and adherend surfaces. Once injected, the grout must flow far into the space and then it must remain in place. Flow is further characterised by ease of injection. Ease of injection is dependent upon even dispersion of filler particles within the emulsion, solids content and the lubricity of the mixture.

Thixotropy

The grout must be thixotropic. Thixotropy is the property of a fluid to display low viscosity under shear force and to set when shear is removed.

Working Time

Working time refers to the time period in which the grout remains in a workable/usable state. It must not set too quickly nor separate out into its constituent components. A desirable working time would be a minimum of 45 minutes to an hour.

Ease of Use and Disposal

The grout should be simple enough to be made on site, require a minimal amount of equipment, and should be easy to clean and dispose. It should be safe both for the applicator and the environment, be low in toxicity and cost (affordable).

Collectively these properties share symbiotic relationships and must be carefully balanced in the design of a treatment proposal. The goal is minimum but opportune intervention towards a reasonable amount of durability and a maximum amount of compatibility. The aim is not to avoid change but rather to manage that which already occurs and induce that which one believes to be beneficial.

4.0 MATERIAL SELECTION

Filled adhesive grouts are comprised of three basic elements, binder, filler, and dispersant (water). Occasionally, an additional element, a thickener is necessary. The formulation of a grout is a balancing of these elements in the correct proportions to achieve the desirable properties as established by the context and the critical performance properties (see 3.3 Treatment Requirements).

As the finish layers at the Casa Grande are clay-based (with lime), an aqueous system was identified as a highly desirable and compatible adhesive option. A water-based grout is fundamentally compatible with the rheological properties of clay allowing wetting and reactivation of both adherend surfaces. Given the criteria of injectibility, two adhesive systems were initially considered: solutions and emulsions. Solutions are a fine even distribution of one substance in another at a molecular level thereby creating a homogeneous mixture in which the separate components are no longer recognizable.[1] The complete dissolution of one material into another offers good depth of penetration, an ideal vehicle for consolidation. In general, the finish layers of the Casa Grande retain good cohesive strength. Consolidation of the matrix is unnecessary. In order to minimize change to the original fabric, the minimum amount of penetration is sought to produce a successful bond between the adhesive and the adherends. Emulsions by their nature are less penetrating than solutions as the solids component within them is essentially immiscible and the resin polymer molecule relatively large. Emulsions were therefore selected over solutions.

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[1] H. Kühn, *Conservation and Restoration of Works of Art and Antiquities* vol. 1, A. Trone trans. (London: Butterworths, 1986), p. 193.

4.1 Adhesive Emulsion

Adhesive emulsion, also known as a dispersion is one of the many forms in which adhesives are produced. An emulsion consists of finely dispersed particles of adhesive polymer in suspension usually in water.[2] These two components are described as the disperse and the continuous phases respectively. In the case of aqueous based emulsions, the immiscible polymer particles are the disperse phase, and water the continuous phase.[3] Various factors contribute to the suspension of these immiscible polymer particles. One factor is the emulsifier, a surface-active agent, used in small percentages which works to keep the particles in suspension. Another factor is *Brownian motion*.

Brownian motion is "the continuous zigzag motion of the particles in a colloidal suspension. The motion is caused by impact of the molecules of the liquid upon the colloidal particles."[4] This constant motion keeps the particles in suspension and prevents them from flocculating or coagulating. Emulsions are able to carry a high solids content in a relatively fluid form, up to 70%, and can be held in a stable suspension at very low viscosities compared with equivalent polymer solutions.[5]
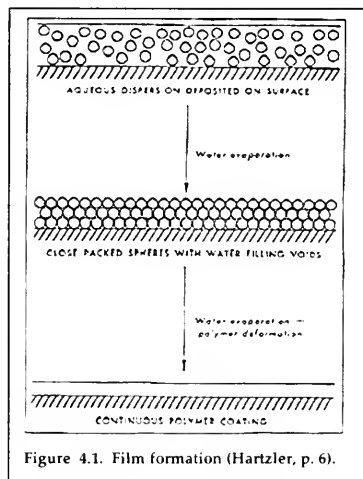
Figure 4.1. Film formation (Hartzler, p. 6).

Common in the construction and packaging industries, adhesive emulsions are typically

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[2] Conservation Unit, *Science for Conservators; Adhesives and Coatings* vol. 3 (London: Conservation Unit of the Museums & Galleries Commission in conjunction with Routledge, 1992), p. 51.

[3] *The Condensed Chemical Dictionary*, 10th ed., revised by Gessner G. Hawley, (New York: Van Nostrand Reinhold, 1981), p. 409.

[4] *The Condensed Chemical Dictionary*, p. 154.

utilized as concrete admixtures and sealants respectively. Adhesives may be categorized by mode of application and setting as either 'thermoplastic' or 'thermoset.' Thermoplastics are predominantly linear macromolecules (polymers) that can be dissolved in solvents and will melt when heated.[5] Thermoplastic resins, such as polyvinyl acetate (PVA) and various acrylates consist of long-chain molecules. In general, the longer the molecule, the stronger the material.[7] Thermoplastic resin adhesive emulsions set by a purely physical, non-chemical process. These emulsions harden by evaporation of their dispersant, water.[8] Once the dispersant evaporates, the particles are forced together to coalesce into a coherent film.[9] Film-forming materials which are useful as coatings, consolidants or adhesives all have large molecular size as a common factor.[10] These polymers do not cross-link during cure so they can be re-softened repeatedly with heat and usually retain their solubility as they age.[11] Their aqueous base and the absence of cross-linking offer a degree of resolubility (reversibility). Thermoplastic resins are widely used in conservation because of the degree of reversibility and stability that they offer.[12]

Thermoset adhesives densely cross-link upon cure and therefore cannot be heated and softened repeatedly. This dense cross linking renders the hard adhesive film insoluble. Curing takes place by chemical reactions at room or elevated temperature depending upon the

[5] C.V. Horie, *Materials for Conservation* (Oxford: Butterworth-Heinemann, 1995), p. 29.

[6] Horie, *Materials for Conservation*, p. 12.

[7] Horie, *Materials for Conservation*, p. 11.

[8] Arthur H. Landrock, *Adhesives Technology Handbook* (New Jersey: Noyes Publications, 1985), p. 129, 181.

[9] Horie, *Materials for Conservation*, p. 29.

[10] Horie, *Materials for Conservation*, p. 29.

[11] Kuhn, *Conservation and Restoration* vol. 1, p. 161.

[12] Constance S. Silver, "Architectural Finishes of the Prehistoric Southwest. A Study of the Cultural Resource and Prospects for its Conservation," 1987, unpublished report. Philadelphia, University of Pennsylvania. Architectural Conservation Laboratory, p. 133.

type. Thermoset adhesives are commonly available as solventless liquids, pastes, and solids.[13] The chemical and physical transformation of thermoset adhesives upon cure and the subsequent insolubility (irreversibility), make these less desirable as a conservation option.

Aqueous adhesive emulsions are made from materials that can be dispersed in water alone. Aqueous based emulsions are lower in cost than the equivalent organic solvent-based systems. In contrast to organic solvent adhesives, their use eliminates health hazards and the danger of fire or explosion. However, in most cases water-based adhesives must be kept from freezing during shipment and storage because of permanent damage to both the container and contents.[14]

4.1.1 Acrylic vs. Polyvinyl Acetate Resin

A search was performed on the history and uses of adhesive emulsions in the field of conservation, with particular regard but not limited to earthen architecture. The questions posed within the search were,

- Type of emulsion (resin)
- Use/delivery
- Concentration
- Context (Location/site)

(See Appendix 10.2 Literature Review of Polyvinyl Acetate and Acrylic Emulsions in Conservation.)

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[13] Landrock *Adhesives Technology*, p. 128.

The oft cited and commonly agreed upon earliest published account of the use of polyvinyl acetate (PVA) in conservation is by Rutherford Gettens and George Stout in 1929.[15] Gettens and Stout utilised polyvinyl acetate as a fixative for the transfer of Oriental wall paintings. Acrylic and polyvinyl acetate emulsions have been used in varying concentrations ranging from 2% to neat (undiluted) for plaster consolidation and reattachment, and adobe amendments according to various published and unpublished accounts. One of the problems encountered in this literature search was the lack of clarity, consistency, and standardisation in specifications. Percentages were frequently mentioned without reference to measure (parts per weight or parts per volume), or to original percent solids as manufactured, and often not specified at all. Ratio interpretations (x:n) varied in meaning from x parts to n parts, to x parts in n parts. Total percent resin available was often never stated. Acrylic emulsions are almost exclusively utilised for exterior applications. Their superiority over polyvinyl acetates for exterior applications was very early remarked by Rutherford Gettens in 1935 and continues to be echoed today as expressed in the current body of literature and in interviews conducted of polymer chemists and conservators for this research. PVAs are regarded as less desirable for exterior environments due to their water and ultra violet light (UV) sensitivity and their low pH (acidity) which decreases (becomes more acidic) over time. They tend to be higher in strength and lower in flexibility compared to acrylics.

> The polyvinyl resins are not a cure for all ills. Their use as an impregnating material for stone surfaces exposed to out-of-door weathering has been suggested and tried, but they have not been found suitable for this purpose because of lack of penetration and the tendency to absorb water, as has already been mentioned.[16]

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[14] Irving Skeist, ed., *Handbook of Adhesives*, 3rd ed., (New York: Van Nostrand Reinhold, 1977), p. 183-185.

[15] Rutherford Gettens, "Polymerized Vinyl Acetate and Related Compounds in the Restoration of Objects of Art," *Technical Studies in the Field of the Fine Arts* IV (1935): 15-28

[16] Gettens, "Polymerized Vinyl Acetate and Related Compounds in the Restoration of Objects of Art," p. 24.

> The superior water resistance of acrylic resins, as compared to polyvinyl acetate, is well known in the masonry field.[17]

Of the acrylic emulsions, Rhoplex® AC-33 (Primal® in Europe and Canada) has been most often selected in conservation for plaster reattachment and stabilisation. Rhoplex® E-330 is most often utilised for adobe and earthen mortar amendment purposes, at least in the United States. It is not clear why Rhoplex AC-33 has been favored by conservators except perhaps for reasons of familiarity. One of the earliest cited usages of AC-33 for conservation purposes is by Giacomo Chiari for the *in situ* reattachment of earthen plasters in Peru, 1975-1977[18] (see Appendix 10.2 Literature Review of Polyvinyl Acetate and Acrylic Emulsion in Conservation). Product specifications of both products reveal almost identical composition and properties (See Appendix 10.3 Product Specifications & Material Safety Data Sheets (MSDS)). During the course of this research, it was discovered that Rohm and Haas, the maker of Rhoplex, plans to discontinue AC-33. Recent research on the history and uses of adhesive emulsions in the field of conservation, an assessment of their performance over time, fulfillment of the desired criteria, and product availability - clearly suggest Rhoplex E-330 as the most desirable candidate for further investigation.[19]

4.1.2 Rhoplex³ E-330

> Rhoplex E-330 Cement Mortar Modifier by Rohm and Haas is a copolymer, a combination of methyl methacrylate and ethyl acrylate, dispersed in an aqueous medium. Created by a process of emulsion polymerization, the

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[17] Morgan Phillips, "Adhesives for the Reattachment of Loose Plaster," *APT Bulletin* 12 (1980): 42.

[18] Giacomo Chiari, "Treatment of Adobe Friezes in Peru," *Third International Symposium on Mudbrick (adobe) Preservation*, p. 39-47.

[19] See Robert Hartzler, *Acrylic-Modified Earthen Mortar*, (Santa Fe: United States National Park Service, 1996).

resulting product consists of a proprietary blend of the two monomers with other minor constituents, with a small particle size, < 1.0μm. These particles are so small that Brownian forces keep them in suspension. Unlike polymers dissolved in solvents (true solutions), polymer emulsions can support high concentrations of polymers of high molecular weight (up to 70%) without becoming unworkably viscous.[20]

Rhoplex E-330 is a milky white liquid, slightly ammonia in odor, containing 47% solids by weight in an alkaline water base. It has a pH of 9.5 - 10.5, and a specific gravity, and melting and boiling points similar to that of water. It will remain stable through five cycles of freeze/thaw at -15° C to 25° C. Its recommended storage temperature range is 1°C to 49° C.[21]

E-330 was designed for the construction industry as an amendment for Portland cement. Rohm and Haas claims that,

> ...cement mortars modified with Rhoplex E-330 are hard, tough, and durable. ...polymerized modified [cement] mortars have superior flexural, adhesive, and impact strengths, as well as excellent abrasion resistance. They possess excellent resistance to ultraviolet light and heat, dry to a uniform color, and offer excellent adhesion to a variety of surfaces, such as concrete, masonry, brick, wood, and metals.[22]

Chief applications of E-330 modified cement mortar include patching and resurfacing, floor underlayments, terrazzo flooring, spray and fill coats, precast architectural building panels, stucco, industrial cement floors, and highway and bridge deck repair.[23]

[20] Hartzler, *Acrylic-Modified Earthen Mortar*, p. 5.
[21] Rohm and Haas, "Material Safety Data Sheet, Rhoplex⁵ E-330," (Philadelphia: Rohm and Haas, 11 July 1990). Water has a specific gravity of 1.0.
[22] Rohm and Haas, "Rhoplex⁵ E-330 Cement Mortar Modifier" (Rohm and Haas Company, 1995), p. 1.
[23] Rohm and Haas, "Rhoplex⁵ E-330 Cement Mortar Modifier," p. 1.

E-330 has been used by the United States National Park Service as an adobe and earthen mortar amendment since 1975. At that time Dennis Fenn ran a series of tests on modified earthen mortars at Chaco Canyon. His work was a continuation of earlier work on E-330 begun by Darrel J. Butterbaugh in 1972. Concentrations of use have varied over time from site to site. As an earthen amendment, E-330 contributes strength, some measure of flexibility and an increased resistance to water the latter extremely important for earthen materials. The increase in hydrophibicity is in direct proportion to a decrease in water vapor permeability. The greater the amount and the higher the concentration of the amendment, the greater the strength and lower the permeability. The emulsion has a minimal affect on the color and texture of the original earthen material and is acceptably stable in exterior environments.

4.2 Binder Concentration

Three percentages of binder concentration were selected for the testing programme, 20%, 40% and neat (undiluted). These concentrations were selected based on the literature review of the use history of adhesive emulsions in conservation applications, the proposed site requirements in this research, and the inherent viscosity of the selected emulsion.

The neat formulation contains the highest resin solids content (47% by weight). In general, viscosity is directly related to resin content whereupon increased resin particle concentration induces higher viscosity due to restricted molecular mobility. Higher viscosity results in

greater surface tension thereby decreasing contact (flow), diffusion and penetration ability.[24] However, what is lost in surface contact is gained in internal film strength (cohesion). Strength, contact, diffusion, and penetration ability are all commensurate with resin solids content. Dilute emulsions raise wettability yet lower strength (adhesive and cohesive). The more dilute the emulsion the lower the percent solids of resin. A 40% binder concentration of E-330 contains 18.8% solids and a 20% binder concentration retains 9.4% solids (by calculation). These figures are critical in understanding the physical effects of resin content alone and in conjunction with a filler fraction (see 4.3 Fillers, below).

4.3 Fillers

"Fillers are added to polymers for many reasons. A filler reduces the volume and cost of polymer used in a formulation."[25] They reduce shrinkage, impart strength and affect viscosity and flow. Microspheres and fumed silica can reduce the weight of an adhesive layer while larger aggregates such as sand contribute strength and hardness. In all cases, fillers decrease the need for the polymer resin content, advantageous in conservation applications. The ideal fillers will be light in weight, aid in shrinkage control and impart strength. In 1980 Morgan Phillips published his findings on filled acrylic emulsion grouts for the reattachment of lime plaster.[26] His system consisted of a combination of reactive and non-reactive fillers, hydrated

[24] "The polymer is not limited in molecular weight since the viscosity does not depend on what is contained within the discrete particles. The viscosity of a latex [emulsion] depends primarily on solids content and the composition of the aqueous phase." Skeist also goes on to mention the relationship between critical surface tensions of adhesive and adherend and the subsequent effect upon wettability. Wettability is the ability of the adhesive to wet the substrate and the ability of the substrate to be wetted. Skeist, *Handbook of Adhesives*, p. 6.

[25] Horie, *Materials for Conservation*, p. 178-179.

[26] See Phillips "Adhesives for the Reattachment of Loose Plaster."

lime, fluid coke, and microballoons. Each filler ingredient contributed towards an ideal particle size range for the application. Matero and Bass combined hydrated lime, ceramic microspheres and fine silica sand for grouts designed for earth-lime adherends.[27] Both formulations utilised hydrated lime for its physicochemical and mechanical compatibility with the original fabric, strength, controlled shrinkage, and its particle size range. In both cases, microspheres were included for their mid-range particle size, lightweight, non-reactivity and the lubricity or injectibility they imparted to the mix. Phillips selected fluid coke for its larger particle size and assistance in shrinkage control as a reactive expansive filler through the generation of CO_2 gas. Matero and Bass chose a fine silica sand as the larger particle size fraction and for the mechanical strength and shrinkage control it offered. These ingredients were selected based on treatment criteria, context and research objectives.

This investigation focuses on inorganic, non-reactive fillers. The selection of fillers was based on previous usage according to conservation literature and the desire to isolate the physical relationships and limits of the various components individually and collectively. Three fillers were selected for testing, fumed silica, glass microballoons, and fine silica sand.

Fumed silica was initially considered a clear choice for a filler because of its ability to fulfill the criteria of thixotropy, low shrinkage, even solids dispersion, compatible strength, injectibility, water vapor permeability, light weight and low cost.[28] Initial tests of emulsions filled with

[27] Frank G. Matero and Angelyn Bass, "Design and Evaluation of Hydraulic Lime Grouts for the Reattachment of Lime Plasters on Earthen Walls," *Conservation and Management of Archaeological Sites* 1 (1995): 97-108.
[28] For other studies of fumed silica as an adhesive filler see Phillips "Adhesives for the Reattachment of Loose Plaster," and Dawn Melbourne, "A Comparative Study of Epoxide Resin and Cementitious Grouts for the Delamination of Sandstone at El Morro National Monument, New Mexico," *Master's thesis, University of Pennsylvania, 1994.*

varying degrees of fumed silica revealed significant problems with cohesive strength. When sufficient amounts of fumed silica were added to the emulsion in order to achieve the desired viscosity, the mixture lost all internal cohesion. Lack of cohesion is synonymous with lack of strength. Fumed silica was eliminated as a filler option for the emulsion concentrations tested.

Quartz sand is an inert common filler known to provide strength and aid in shrinkage, however sand alone does not meet the required criteria of injectibility and minimal weight. A combination of sand and microspheres was identified as a possible option.[29] The combination of two fillers introduced a new ratio of solid to solid (microspheres to sand). A range of ratios was specified in order to isolate the contribution of individual solid components and thereby determine the effects of each solid on the character of the mixture in relation to the binder and to one another (see Table 5.1 Test Sample Matrix).

The particle size range of the sand ($\leq$300μm) was selected to pass through a 16 gauge needle, the necessary orifice to deliver the grout in the Casa Grande detachment gap. The sand contributes strength by its large particle size, surface area and sub-angular shape. Microspheres offer lightweight and lubricity due to their hollow spherical form and lend body to an emulsion, augmenting viscosity thereby decreasing the need for resin beyond what has been determined to be sufficient for bonding strength and thickener. Glass microballoons were selected over ceramic microspheres for their availability in smaller particle size (30-110μm). The extended particle size range of the solids fraction offers greater strength and shrinkage control. Ideally, the correct combination of these fillers will reduce the need for

resin by providing some of the internal physical structure otherwise provided by the polymer molecules. The filler fraction contributes infrastructure to the grout through a measure of mechanical bonding and increased surface area or points of contact onto which the polymer molecules can attach themselves. A reduction in resin means a lower binder concentration, which is critical in reducing strength and increasing water vapor permeability. Clearly, a balance must be struck in identifying the optimum resin content (as available solids) necessary for strength without imparting water vapor impermeability.

The ideal grout formula lies in a balance of liquid to solid (binder to filler) and solid to solid (microballoons to sand) so that the stated criteria are achieved: good injectibility, flow, minimal shrinkage and weight, good adhesion, bond strength and cohesion, reasonable water vapor permeability, and good stability

4.4 Thickeners

The function of thickeners is twofold: one, it augments the viscosity, and two, it is critical in maintaining suspension and even dispersion of the solids fraction. Laboratory and field tests revealed that without the thickener, the filler solids fraction quickly separated out from the binder and from its own constituent components (microballoons from sand). Only one thickener was selected for use, Acrysol[5] 6038A. Acrysol 6038A is a sodium polyacrylate thickener recommended by Rohm and Haas as a rheology modifier (viscosity) for use with Rhoplex AC-33 and other latex compounds. It is produced in a gel form, and is relatively clear

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[29] This consideration of this combination was based on previous research conducted by Matero and Bass. See

in color. It has a pH of 8.0 - 9.5, a specific gravity of 1.0 - 1.2 and a viscosity of 18000 - 25000 (as is, cps Brookfield RV, spindle #6, 20 rpm, 25° C). It is considered stable, and recommended for use within the temperature range of 1° - 49° C.[30] It is dilutable in water and described as "especially stable to viscosity drift with time in a wide variety of compounds."[31] Undiluted Acrysol 6038A contains 13 - 13.5% solids. The manufacturer recommends cutting this with water at a 1:1 ratio for easier incorporation into the emulsion system. This concentration yields an approximate solids content of 6.5%.[32]

"Design and Evaluation of Hydraulic Lime Grouts."

[30] Rohm and Haas, "Material Safety Data Sheet Acrysol® 6038A Emulsion," 1998.

[31] Rohm and Haas, "Acrysol® 6038A," product literature, 1992.

[32] Temperature had a distinct affect upon the thixotropy and dispersion of the grout formula. The average ambient laboratory temperature during the testing program was 25° C. The average temperature on site at Fort Davis National Historic Site where the grout was field tested during the summer of 1998 following the laboratory test program, was 35° C. In the laboratory a 1:2 ratio of Acrysol:H₂O (approximately 4.3% solids) was found to be a workable concentration. This concentration was easily measured and distributed through a hypodermic syringe and was dilute enough so as not to drastically alter the inherent characteristics of the grout formulation. Thickener was used as an enhancer not as a base ingredient. The high temperatures experienced on site raised the viscosity and thixotropy of the grout. The grout set much more quickly in the high heat resulting in an impractical short working time. The thickener was still necessary for even solids dispersion however not at such a high concentration. The Acrysol ratio was decreased to 1:2.5 Acrysol:H₂O (approximately 3.7% solids) in response to the uncontrollable high heat experienced in the field.

5.0 TESTING PROGRAMME

A laboratory testing programme was developed to qualitatively evaluate the effect of each individual variable on the properties of the grout system: injectibility, flow, shrinkage, solids distribution, and strength. Variables included:

- binder concentration (% solids)
- liquid to solid component ratio (L:S) and
- filler fraction, microspheres to sand (M:S).

One hundred different formulae were tested, 96 of which are included in the following test matrices. The four excluded formulae included reactive ingredients of lime and fluid coke. These recipes were based on Morgan Phillips's 1980 and 1986 research and were immediately eliminated as they failed to fulfill the initial requirement of injectibility as defined for this particular usage at CAGR.[1]

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[1] Morgan Phillips published his work on filled acrylic emulsions for the reattachment of lime plasters to wood lath for the first time in 1980 and again in 1986. (See Phillips "Adhesives for the Reattachment of Loose Plaster," and "Experiences in the Use of Acrylic Plaster Adhesives" *Case Studies in the Conservation of Stone and Wall Paintings* N.S. Brommelle and Perry Smith eds. (London: International Institute for Conservation of Historic and Artistic Works, 1986), p. 34-37. In his work, two types of adhesive binders were employed, Rhoplex MC-76 and LC-67. These were used together or separately depending on degree of stiffness and flexibility desired. He utilized these binders in an unfilled state when the space between lath and loosened plaster was less than 3 mm. Phillips used a thickener to impart thixotropy and increase viscosity for both filled and unfilled formulations. In 1980 the recommended thickener was fumed silica, by 1986 Phillips had replaced the fumed silica with an acrylic polymer gel, Acrysol ASE-60. This he found to be far more compatible in an aqueous system and now recommended it exclusively. For fillers Phillips used a three part system comprised of a combination of dry powdered hydrated lime, for the fine particle size fraction, glass microspheres for the intermediary particle size fraction, and fluid coke for the large particle size fraction. His method of application was injection through a nozzle with an exterior diameter of 4 mm which was "sufficient to allow reliable passage of the filled adhesive." His ports of entry were 5 mm in diameter as drilled. The treatments were applied in a sheltered architectural interior environment.

In his 1986 article, Phillips offers two formulae for filled adhesives, one for stiffer, the other for more flexible applications. The more flexible formula was replicated here for section 5.1 Flow of the testing program.

     2 parts Microballoons
     2 parts lime
     2 parts coke
     3 parts acrylic emulsion
     1/3-1/2 part water

The different grout formulations were designed to expose the limits of individual components within a given property. What effect does a higher proportion of sand have on flow? What effect does a higher concentration of polymer solids have on strength and water vapor permeability? The range of grout formulations provides a relative reference guide to understanding the effects that each component produces. Three concentrations of binders were tested, 20% (9.4% solids), 40% (18.8% solids) and neat (N) (undiluted, 47% solids) v/v (e.g. 20% = 20 ml binder + 80 ml H_2O). Four ratios of liquid to solid [binder (resin and water) to filler (microspheres and sand)] were tested, 1:1; 1:2; 1:3; and 1:4 by volume (e.g. 1:3 = one part liquid to three parts solid). Eight solid fractions were tested to determine the ideal ratio of microspheres to sand (M:S).

Liquid:Solid / Binder Concentration

		1:1			1:2			1:3			1:4		
S		**M:S**	**20%**	**40%**	**N**	**20%**	**40%**	**N**	**20%**	**40%**	**N**	**20%**	**40%**	**N**
o	**F**	**1:0**	1	2	3	4	5	6	7	8	9	10	11	12
l	**i**	**0:1**	13	14	15	16	17	18	19	20	21	22	23	24
i	**l**	**1:1**	25	26	27	28	29	30	31	32	33	34	35	36
d	**l**	**2:1**	37	38	39	40	41	42	43	44	45	46	47	48
:	**e**	**3:1**	49	50	51	52	53	54	55	56	57	58	59	60
s	**r**	**3:2**	61	62	63	64	65	66	67	68	69	70	71	72
o	**s**	**4:1**	73	74	75	76	77	78	79	80	81	82	83	84
l		**5:1**	85	86	87	88	89	90	91	92	93	94	95	96
i														
d														

Table 5.1 Test Sample Matrix

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thickener as required

Phillips notes that this formulation produces a filled adhesive foam designed for injection application with a caulk gun with a 3-4 mm interior diameter nozzle. This is three to four times the size of a 16 gauge needle used in the present research programme. The above formulation was attempted with both neat and 40% binder concentrations,

The testing programme was ordered hierarchically beginning with the most critical or fundamental physical requirements and progressing on to more complex relationships. If a formulation could not fulfill the primary requirements of injectibility and flow then it was immediately eliminated from further testing. Formulations were tested for flow, shrinkage, shear strength under compressive force, and water vapor transmission, in that order. The predefined ideal mixture directed the process of elimination. The first stage of evaluation (and elimination) was based on physical performance during testing. Those mixtures that clearly, visibly failed to satisfy the properties being tested were immediately eliminated. The second stage of evaluation was based on theoretical preferences and history of performance as supported by research. The third stage of evaluation was based on research limitations of time and available resources. In testing research, it is important to test a range of options that offer useful points of reference. The points of reference are initially determined by the stated

Figure 5.1. Grout materials (Fong, 1998).
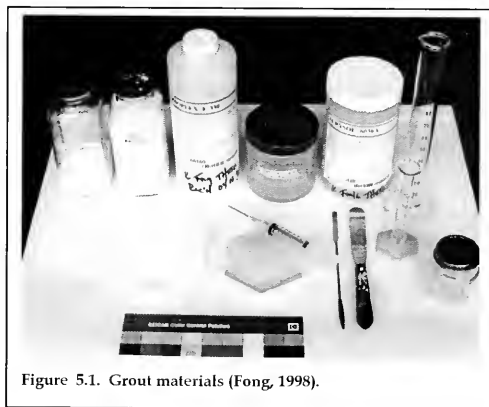
neither was injectible. This failure to fulfill the first criteria of injectibility revealed that further research was necessary in the area of filled acrylic emulsions. Phillips's formulae were eliminated from further testing.

solution. These reference points are reevaluated and redefined as necessary after each phase

of testing based on test results.

Basic grout fabrication materials were the same for each test for reproducibility. Additional

equipment was included as needed for each specific test.

Materials[2]

Liquid:	Rhoplex E-330 (20%, 40% and N)
Fillers	
Microspheres (M)[3]:	3M Scotchlite Glass Bubbles K20 (30-110 µm)
White quartz sand (S):	$\leq$ 300µm.
Thickener:	Acrysol 6038A (1:2, Acrysol:deionized H_2O)

Equipment

    syringes; 10 cc, 20 cc
    B-D 16G 1 1/2 Precision Glide' needles
    micro-spatulas
    small containers with lids for mixing and storing formulae
    graduated cylinders; 10 ml, 100 ml

All grout formulations were hand blended by either agitation or stirring. The less viscous

formulations were easily mixed by hand shaking of the sealable container in which they were

made. The more viscous formulations were hand stirred until thoroughly blended. The order

of manufacture was as follows,

- measure and dispense liquid portion into container

- measure and combine solids fraction in separate container

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[2] All ratios are measured by parts per volume (ppv).

[3] During the course of the testing program the author developed a severe epidermal allergic reaction requiring medical treatment. The reaction resulted in a highly agitated and extremely itchy blistering rash. The irritant was believed to be the glass microballoons. The product MSDS sheet warns against prolonged exposure and contact, which could lead to irritation of the skin, eyes or throat. The author developed an intolerance and hypersensitivity to the material after two months of near constant laboratory exposure. This is an extreme amount of exposure. With proper protection, precautions, and limited and reasonable exposure no other individuals experienced any adverse reactions to the material. The reaction took a full three months to completely disappear.

- pour solids fraction into liquid fraction

- mix thoroughly to the point of even distribution

- add thickener as needed and mix thoroughly

5.1 Flow

Purpose

This test was designed to measure the properties of flow and injectibility. One hundred grout formulations were tested. 1 cc of each grout formulation was injected with a hypodermic syringe through a 16 gauge needle onto a vertically positioned wood board. Wood was used due to its semi-absorbent surface in simulation of the water absorbency of the earthen adherends. Formulations were eliminated based on their failure to,

- pass through the needle, and

- poor flow.

Poor flow was defined as either,

- too high resulting in failure to flow down the board to at least 2.5 cm the vertical distance,

- too low yielding an uncontrolled flow of liquid down the board to at least 9.5 cm the vertical distance.

The flow test was conducted in conjunction with the shrinkage tests for expediency's sake. One batch of grout formula was easily distributed between the two tests.

Additional equipment
   wood board

Methodology

Twenty cc of liquid was measured into a small glass container. In a separate, dry container

fillers were measured, combined and then added to the liquid. The jar was vigorously shaken
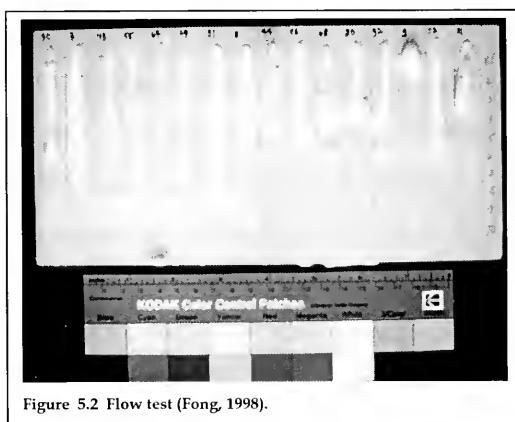
Figure 5.2 Flow test (Fong, 1998).

for a few minutes until all contents were evenly distributed and dispersed. This method of

shaking was sufficient for groupings (L:S) 1:1 and 1:2 only. Grouping 1:3 was hand stirred

due to the ratio of L:S. Once fillers were evenly distributed throughout liquid, thickener was

added as needed. Thickener was measured with and distributed by syringe. Mixture was

then hand stirred until thickener was thoroughly distributed (in these amounts, approximately

1.5 – 2 minutes). Grout was extracted from the container with a syringe, turned nozzle up and

tapped to raise and release trapped air. The needle was attached and one cc was extruded

onto the board for the viscosity drip test. Flow was measured as a function of the vertical

distance traveled. This marked the preliminary criteria of injectibility. Any formula that failed to pass through the needle was automatically rejected.

5.2 Shrinkage

Purpose

An emulsion sets to a film, first by loss of the dispersant, (with this case water), and then by coalescence of the small polymer particles.[4] Evaporation and absorption of the dispersant are the processes by which shrinkage occurs. Shrinkage frequently produces cracks and deformation of the material and is here regarded as shrinkage failure. Two phases of shrinkage tests were performed. The first preliminary phase was conducted in hot glazed ceramic spot plates to reduce test numbers and equipment. The non-porous, hydrophobic surface of the glaze and the concave form of the well influenced the process of evaporation and consequent shrinkage patterns. In the process of setting, the water dispersant evaporated most rapidly from the top surface perimeter or the shallow sides of the well, while the center retained water for a longer amount of time. This uneven set produced a characteristic pattern of central collapse (referred to in the test observations as 'sink holes') for some formulations. Only those formulae deemed most successful (low shrinkage) after phase one were promoted to the second phase of shrinkage tests.

The purpose of the second (confirmatory) phase of shrinkage tests was to more accurately replicate the process of shrinkage and setting on porous adherends by utilising unglazed,

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[4] Horie, *Materials for Conservation*, p. 5.

porous ceramic flat-bottomed molds. This closer approximation to the actual context produced more site relevant reactions between the grout and its adherends. Terra cotta flowerpot saucers were soaked overnight in water in order to prevent rapid drying through suction of the grout mixtures in the porous molds. Saucers were hand dried with paper towel, slowly filled with 10 cc of grout and allowed to set, air drying in the laboratory.

Additional equipment
        Glazed ceramic spot plates; well dimensions - top diameter 2.7 cm, depth 1 cm
        Terra cotta saucers; base diameter 4 cm, depth 1.5 cm, top diameter 5.5 cm

Methodology

Phase One - Spot Plates

Grout preparation and injection procedure was the same for all tests. 4 cc were extruded into individual spot wells, excess was screed with an even edge, and disks were allowed to air dry set in the laboratory.
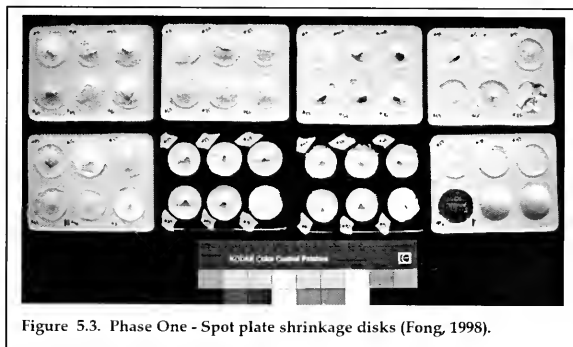

Figure 5.3. Phase One - Spot plate shrinkage disks (Fong, 1998).

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[4] Horie, *Materials for Conservation*, p. 5.

Phase Two – Unglazed Ceramic Dishes

Terra cotta saucers were soaked overnight in water to prevent rapid drying of emulsion mixtures into the porous molds. Saucers were hand dried with paper towels. 10 cc were injected into the individually labeled ceramic saucers and allowed to air dry cure in the laboratory.

Figure 5.4. Phase Two - Ceramic dishes (Fong, 1998).
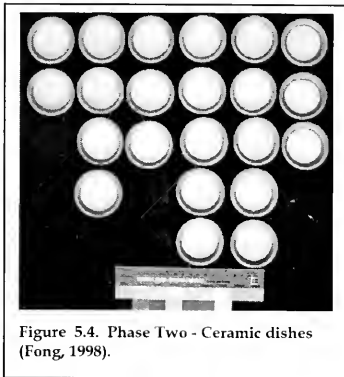
5.3 Dispersion

Dispersion refers to the even distribution of the solids fraction. Do the solids remain in suspension during application and set/cure? Other characteristics related to dispersion are

Figure 5.5. Cut ceramic disks for dispersion investigation (Fong, 1998).
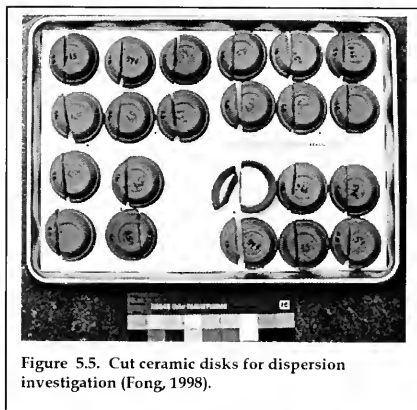
internal cohesion and porosity. A cross section of each phase two shrinkage test sample was cut for qualitative analysis. Samples were cut with a diamond edged masonry circular saw blade, sanded, and trimmed. All samples were inspected visually and with a Nikon SMZ-U under quartz halogen reflected light illumination at 10x magnification.

5.4 Shear Strength Under Compressive Force

Purpose

To measure adhesive bond strength and reveal point of failure. The ideal point of failure is within the adhesive itself or at the interface between adhesive and adherend, but never within the adherend. ASTM D3931 - Standard Test Method for Determining Strength of Gap-filling Adhesive Bonds in Shear by Compression Loading was used as a model for assembly fabrication and test method.[5]

Additional materials

Release agent:	Mineral oil
Assemblies:	Site soil; original fabric $\leq$ 2.36 mm particle size
	3 mm x 3 mm bass wood spacers
	Plasteline modeling material
	Duco cement
	Rags

Additional equipment

Wooden molds 9 cm x 9 cm x 2.5 cm[6]
Plastic wrap
Wood board
1/4" x 1/4" wood strips
Cotton
Plastic straw or other similarly sized cylinder

Methodology

Nineteen assemblies were produced from the original Casa Grande wall loss fragment.[7] The dense caliche was rehydrated for 24 hours, mechanically broken down by hand, air and heat

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[5] "D3931, Standard Test Method for Determining Strength of Gap-filling Adhesive Bonds in Shear by Compression Loading," (Philadelphia: ASTM, 1990). Modified by author.
[6] Coupon dimensions are modified from original test dimensions.
[7] A section of the 1995 exterior wall loss fragment from the Casa Grande was sent to the ACL/UPenn, for research and testing. Available quantity of the wall loss fragment dictated that six different formulae could be tested for strength and water vapor permeability. These six were selected based on their ability to meet the three previously tested criteria of flow, shrinkage, and dispersion.

lamp dried, and further divided by hand. Once the material was completely dry it was crushed in a mortar with a rubber covered wooden pestle. The reconstituted crushed material was sieved through a no. 8 sieve, $\leq$ 2.36 mm.
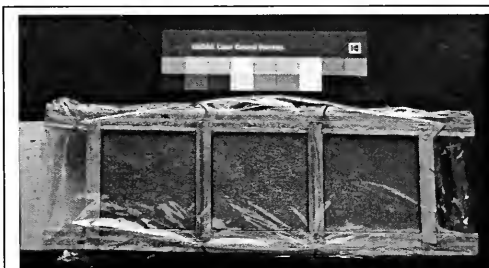
Figure 5.6. Strength test block fabrication, in mold (Fong, 1998).

That which passed was used for coupon fabrication. Through this method, the very large ($\geq$ 2.36 mm) caliche nodules were removed from the dry mix material. This particle size range was selected according to earlier material characterisation data for the finish layer, (see section 2.3.4 Thin Section Analysis). Wooden molds were soaked for two hours in mineral oil, excess oil was removed with paper towels and molds were placed on a flat wooden board. Dry sieved earthen material was combined with deionized water and mixed according to ASTM C305, Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency.[8] A

Figure 5.7 Strength test block fabrication, tented in molds (Fong, 1998).

minimum amount of water was used to achieve a workable consistency. Less water reduces shrinkage yet the mixture must to be pliable enough to be molded. The rough ratio of dry material to water was 3.5:1 (soil: water) by weight. The mixture was worked into molds with

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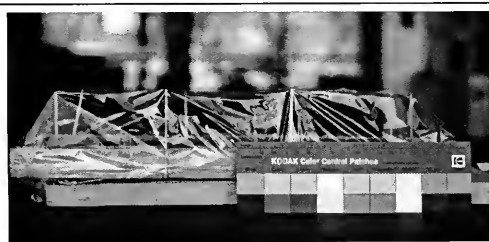a plastic spatula, the board was tamped to release air from the blocks, and the excess was screed from the tops of the blocks. Molds were tented with plastic wrap for two days at which point a straw was inserted into the center of half of the blocks to create an injection port hole for the grout formula application. Molds were removed after two days or once blocks had shrunken sufficiently to retract from molds, whichever came first. Blocks remained tented for another two days. Blocks were turned over and relocated on the board to dry areas and the plastic wrap was removed. Blocks were air cured in the laboratory until dry (average May laboratory temperature was $28°$ C, average relative humidity (RH) was 60).
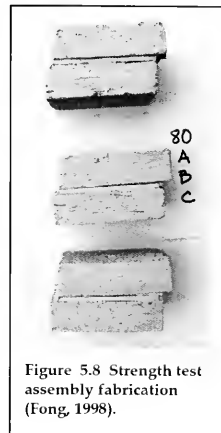
Figure 5.8 Strength test assembly fabrication (Fong, 1998).

Assemblies were fabricated based on guidelines provided in ASTM D3931. Three assemblies were made per formula (A, B, C) plus one additional for machine calibration for a total of 19
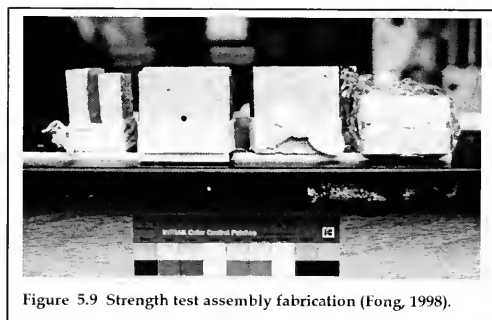
Figure 5.9 Strength test assembly fabrication (Fong, 1998).

assemblies. Three mm² bass wood spacers were placed on three sides of the smooth face of the unpunctured coupon and tacked down with a few drops of Duco cement, just enough to hold the spacers in place. This distance replicated the typical gap of

[8] "C305, Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency," (Philadelphia: ASTM, 1987). Modified by author.

Type A detachment. The punctured coupon was placed atop the coupon with spacers, smooth side facing spacers (smooth face to smooth face), and offset by 1/4 inch. Coupon sides with spacers were sealed with Plasteline modeling material. The top edge was left open for visual access and air displacement during grout injection. The assembly was further secured by a fabric swath tied in a secure knot beneath the port hole.

Grouts were made in batches of 140 cc. These larger quantities required less amounts of thickener proportionately compared to the smaller batches produced for the flow and shrinkage tests. After five minutes in a sealed container, the surface of the grout formulae solidified yet all easily reliquified with active stirring, (a demonstration of thixotropy).

Grout application followed standard methodology of prewetting with a dilute solution of the emulsion, here a 10% concentration of E-330 in water. Prewetting is a critical step in the treatment process. It prepares the adherends by reducing suction/rapid absorption, decreasing friction thereby increasing flow and adhesion of the grout, and subsequently aids in controlling shrinkage. Prewetting with a dilute solution of emulsion creates a bridge/key between the grout and the adherends. The low (4.7% solids) resin content of the 10% concentration provides low viscosity, and consequent rapid distribution and absorption. However, the polymer molecule size remains the same and it is the size of the molecule and the porosity of the adherend that limit the molecule's penetration. In this specific case, deep penetration of the resin was unnecessary. The original building fabric is extremely dense and in no need of consolidation. Minimal penetration provides a key into the surface of the adherend. The porthole of each assembly was plugged with Plasteline and the gap filled from

the open top edge, with the prewetting solution. As the 10% concentration was absorbed into the adherends, the gap was refilled to the top edge. To insure thorough prewetting, the solution was allowed to sit in the assembly for approximately five minutes after which time the remaining liquid was emptied from the gap.

Once the prewetting stage was complete, the grout mixture was injected through the port hole with a hypodermic needle and syringe. Cotton was held around needle and porthole to block reverse airflow while the grout was slowly injected into gap. The needle was periodically turned to shift flow direction given its bias cut. The opening at the top of the assembly allowed visual access to grout flow within the gap. Once the gap was filled or ceased to receive grout, the excess was screed from the top edge, the assembly was wrapped in plastic
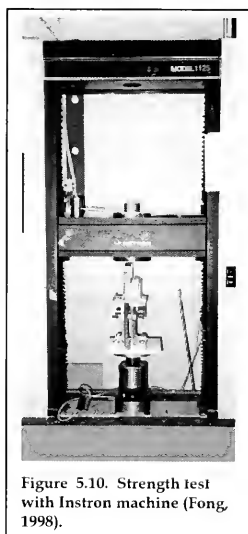
Figure 5.10. Strength test with Instron machine (Fong, 1998).

and set aside to cure for two days (vertically orientated, in order to more accurately simulate the site conditions). After two days, individual wrappings were removed, and assemblies were retented with plastic wrap in groups of six. In this way, air circulation was gradually increased and moisture decreased. Assemblies were allowed to air dry (set) in the laboratory over a period of two weeks (average May laboratory temperature was 28° C, average relative humidity (RH) was 60). Fabric bindings were removed at point of set.

Strength tests were conducted with the assistance of Dr. Alex

Radin at the Laboratory for Research on the Structure of Matter (LRSM) at the University of Pennsylvania. Tests were performed with an Instron Testing Machine Model 1125 with a cross head speed of 0.1 inches per minute and a chart speed of 5 inches per minute. The machine was calibrated to accommodate the form and characteristics of the assemblies,
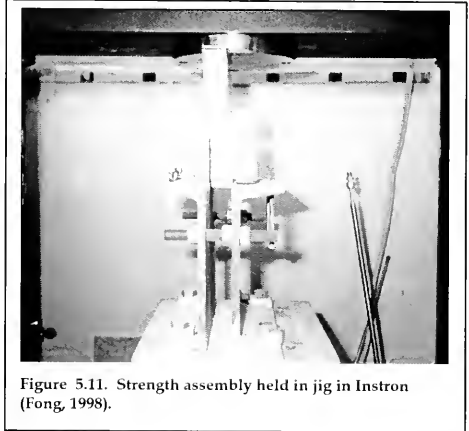
Figure 5.11. Strength assembly held in jig in Instron (Fong, 1998).

(dimensions and strength of caliche material being tested). Each sample was placed in the jig. A squared, metal rectilinear block was placed atop one coupon of the assembly. This was the surface upon which vertical compressive force was exerted. The metal block covered the entire top cross section area of the coupon. The base surface of the other assembly coupon was the bottom cross section area of the parallel resting block. It was essential that these block surface areas of contact did not include any surface area of the wooden spacers or the strength of the adhesive fill would not be isolated. If the metal block was not parallel with the crosshead, shims were placed between the two surfaces to more evenly distribute the compressive force across the coupon cross section surface area.

5.5 Water Vapor Transmission (WVT)

Purpose

To assess the relative water vapor permeability of a 3 mm layer of the grout on the earthen adherend. The ideal formulation is that with the highest or most similar WVT to that of the original fabric. ASTM E96 - Standard Test Methods for Water Vapor Transmission of Materials and NORMAL 21/85 Water Vapor Permeability were used as guidelines in the assembly fabrication, data calculation and interpretation of the test.[9] NORMAL defines water vapor permeability as,

> The amount of water vapor, per unit time and surface, that flows through a material of parallel surfaces and a given thickness due to a water vapor pressure difference between the two surfaces.[10]

Additional materials
     Assemblies: Site soil; original fabric $\leq$ 2.36 mm particle size

Additional equipment
     pvc pipe section molds, 7 cm interior diameter x 2 cm height
       disk diameter[11] = 6.7 cm
       disk area = 35.27 cm^2
     electrical tape
     wax
     250 ml beakers
     lab counter paper (one side absorbent, one side hydrophobic)
     plexi-board or other hydrophobic surface
     Drying rack
     desiccation chamber
     desiccant
     cotton

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[9] "E96, Standard Test Methods for Water Vapor Transmission of Materials," (Philadelphia: ASTM, 1996) and "NORMAL, 21/85 Water Vapor Permeability," draft translation photocopy, Architectural Conservation Laboratory, University of Pennsylvania.

[10] "NORMAL, 21/85 Water Vapor Permeability."

[11] As per NORMAL 21/85 and ASTM E96, disks were formed to a uniform thickness at least five times greater than the deepest pit size and three times the thickness of the largest grain.

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thermohygrometer
scale sensitive to 0.01 (g)

Methodology

The caliche preparation was identical to preparation in the shear strength assembly fabrication

(see 5.4 Shear Strength Under Compressive Force). A total of 19 disks were fabricated from

the original wall loss fragment to test six grout formulations; three disks per formulation and

one untreated control. PVC pipe section molds produced disks with 6.7 cm diameter, 2 cm

thickness and 35.27 cm^2 surface area. Once dry, the exterior edge was wrapped in electrical

tape. The tape extended 3 mm above
the smooth face of the disk to serve as
a retaining wall for the 3 mm film
layer later to be applied. Disks were
prewet with 6-7 cc, up to the fill line,
of 10% concentration of E-330. Once
absorbed, 12 cc of grout were injected
onto each disk to form a 3 mm layer.
Disks were set aside to air set for one
week.
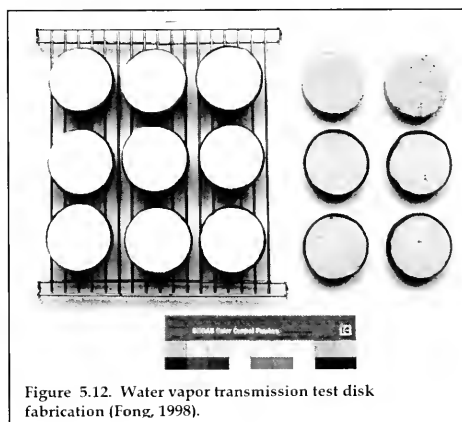
Figure 5.12. Water vapor transmission test disk
fabrication (Fong, 1998).

Disks were sealed in the openings of 250 ml beakers each containing 140 ml of deionized H$_2$O

and placed in a closed glass chamber containing calcium sulfate desiccant. The chamber and

desiccant were monitored to create an environment of a relative stable humidity as defined by

ASTM E96. Assemblies were removed from the chamber and weighed and their positions
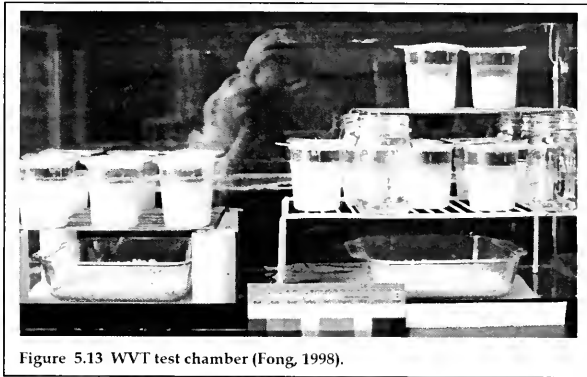
Figure 5.13 WVT test chamber (Fong, 1998).

rotated within the chamber once every 24 hour cycle. The test was run over a period of 17 days to insure a steady state of weight loss was achieved.

6.0 TEST DATA RESULTS AND OBSERVATIONS

The testing programme was designed to isolate specific aspects and properties of each grout formulation. Standard tests such as those provided by ASTM were often adapted in order to accommodate the tested materials, here earth and filled acrylic grout, and the desired properties. Where tests did not exist, they were created or adapted from others, such as those used for flow and shrinkage. The limitations of material resources (original caliche building material) and time limited the precision, accuracy and absolute reliability of the test data however general qualitative and approximate quantitative conclusions were possible.[1]

The greatest strength of a well-conceived testing programme is its ability to measure the inherent properties of the material as well as reveal inaccurate assumptions regarding those characteristics and attributes. This is achieved through a rigorous, methodical programme that produces cross-referential and reproducible data. Results of the combined tests revealed inherent characteristics, which were then interpreted as strengths and weaknesses, and collectively corroborated or challenged existing hypotheses. This process directed each subsequent phase of investigation. The flow and shrinkage tests performed this function vividly. Observations of unacceptable levels of aeration or speculation on disproportionate levels of sand in one test were frequently confirmed in the other. This cross referencing of data resulted in more thorough and accurate conclusions.

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[1] Assemblies for the strength and water vapor transmission tests were made of earthen material from the site. A limited amount of this material was offered for research use from CAGRNM. The amount of material available dictated the number of assemblies produced. This in turn, dictated the number of mixtures which could be tested.

6.1 Flow[2]

Liquid:Solid (L:S) / Binder Concentration

	1:1			1:2			1:3			1:4		
M:S	20%	40%	N	20%	40%	N	20%	40%	N	20%	40%	N
1:0	1	2	3	4	5	6	7	8	9	10	11	12
0:1	13	14	15	16	17	18	19	20	21	22	23	24
1:1	25	26	27	28	29	30	31	32	33	34	35	36
2:1	37	38	39	40	41	42	43	44	45	46	47	48
3:1	49	50	51	52	53	54	55	56	57	58	59	60
3:2	61	62	63	64	65	66	67	68	69	70	71	72
4:1	73	74	75	76	77	78	79	80	81	82	83	84
5:1	85	86	87	88	89	90	91	92	93	94	95	96

Shaded areas indicate those formulae deemed unsuccessful.

Table 6.1 Flow Test Sample Matrix

If a formulation clearly failed to exhibit the necessary flow properties as defined it was eliminated. Those that displayed acceptable or hopeful flow properties were promoted to the next series of tests.

General Observations

Viscosity is in direct proportion to solids content of both the polymer and the filler. Too low of a filler fraction produced uncontrollable low viscosity. Too high of a filler fraction produced an uninjectible paste. The combination of a high polymer concentration (N) with a high filler solids fraction produced the same uninjectible result.

Sand is clearly a necessary component for strength, however the inclusion of too much sand produced heavy grouts. Microspheres contribute lubricity and suspension and aid in the even

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[2] The flow and shrinkage tests were performed concurrently. Failure in the flow test was noted independently of the shrinkage test. The results of the two tests were subsequently compared and analysed for corroborating or conflicting data and formulae selected for the next round of tests.

dispersion of solids. A low microsphere to sand ratio lacked the necessary support to carry and distribute the sand fraction. Too high a microsphere fraction resulted in poor strength.

Aeration is in direct relation to the filler fraction. Formulae with low solids contents (1:1 and 1:2 L:S) exhibited high aeration during mixing manifested as a thick layer of foam. Aeration became air entrainment with the incorporation of thickener. A noticeable decrease in aeration was observed at the 1:3 L:S grouping. High air entrainment increases porosity and decreases weight, but it greatly reduces strength. The level of aeration visible in the 1:1 and 1:2 groupings was undesirable.

The amount of thickener required to achieve a desired viscosity was most directly related to binder concentration. Thickener amount was relatively consistent within a category of binder concentration within an L:S grouping. Within any L:S grouping, the lower the concentration of binder, the greater the amount of thickener necessary to achieve the desired viscosity. However, the filler content of the L:S ratio also affected the thickener fraction. This became apparent in the 1:3 L:S grouping where the higher filler fraction required less thickener than the 1:1 and 1:2 groupings and the 1:3 L:S, Neat category required the least amount of thickener of all categories. The thickener fraction was a result first, of binder concentration (percent solids) and second, of L:S ratio. M:S, solid:solid ratio had little noticeable affect on the thickener fraction.

conflicting data and formulae selected for the next round of tests.

81

Liquid:Solid Ratios (L:S)

L:S 1:1

The low solids content in this grouping resulted in a high level of aeration and an uncontrollable low viscosity. The low 20% binder concentration coupled with the low filler solids content produced an irreconcilably low viscosity. The 20% category was eliminated from further testing.

L:S 1:2

Formulae from this grouping still displayed undesirable levels of aeration. Viscosity was slightly improved due to the increase in filler solids, especially at the 40% binder concentration level however the degree of aeration compromised the strength properties. As with L:S 1:1, the 20% binder concentration category produced an uncontrollable low viscosity. The 20% category was eliminated from further testing.

L:S 1:3

This category produced the most promising results. The increase in filler solids dramatically decreased aeration and increased viscosity to a desirable level, including the 20% binder concentration category.

L:S 1:4

The ratio of filler solids to binder content was too high producing an uninjectible paste. This grouping was immediately eliminated from further testing.

Filler Ratios, Microspheres:Sand (M:S)

The 1:0 M:S category revealed that sand is a necessary component for strength. Dried rivulets of this solid:solid ratio all easily yielded beneath fingernail pressure. The 0:1 and 1:1 categories revealed that the sand content must be a fraction of the microsphere content in order to remain in suspension and/or pass through the needle. The 2:1 and 3:2 categories further revealed that the sand fraction must be less than 2/5 of the total filler content. The 20% and 40% formulae of these M:S filler fractions, while injectible produced heavy mixtures which quickly ran down the board due to their weight thereby failing to fulfill the stated ideal properties of appropriate strength and minimum weight. The combination of a high polymer solids content (N) with the 2:1 and 3:2 M:S sand fraction in the 1:2 and 1:3 L:S groupings produced uninjectible mixtures, (42, 45, 66 and 69).

Binder Concentration (%)

All 20% binder concentration formulae in the 1:1 and 1:2 L:S categories displayed unacceptably low viscosity levels. These formulae were eliminated from further testing. Lower filler fractions require higher concentrations of polymer solids in order to provide the overall solids content and adhesion necessary for viscosity and strength. A 1:3 L:S ratio contained a high enough filler solids content to tolerate a low binder concentration.

6.2 Shrinkage

There is a definite relationship between percentage of solids and shrinkage. The higher the percent solids (both filler and polymer), the less shrinkage occurs. The highest degree of shrinkage was always found in the 20% binder concentration range. This is attributable to the low resin, high water content. The higher the water content, the greater the shrinkage due to the higher percentage of evaporation and volume change. Similarly, the higher the liquid ratio, the greater the water content the higher the percentage of evaporation.

Phase One Preliminary - Spot Plates[3]

Liquid:Solid (L:S) / Binder Concentrations

		1:1			1:2			1:3			1:4	
M:S	20%	40%	N	20%	40%	N	20%	40%	N	20%	40%	N
1:0	1	2	3	4	5	6	7	8	9	10	11	12
0:1	13	14	15	16	17	18	19	20	21	22	23	24
1:1	25	26	27	28	29	30	31	32	33	34	35	36
2:1	37	38	39	40	41	42	43	44	45	46	47	48
3:1	49	50	51	52	53	54	55	56	57	58	59	60
3:2	61	62	63	64	65	66	67	68	69	70	71	72
4:1	73	74	75	76	77	78	79	80	81	82	83	84
5:1	85	86	87	88	89	90	91	92	93	94	95	96

Bold numbers indicate formulae tested during this phase.
Shaded areas indicate those formulae deemed unsuccessful.
Table 6.2 Shrinkage Phase One Test Sample Matrix

General Observations

Characteristic patterns of shrinkage occurred as 'sink holes' and surface cracks. A sink hole is defined as a hole in the center of a disk or central collapse. This observed shrinkage phenomenon may be due to the affect of the physical form and material of the disk molds – hot glazed porcelain, wet chemical spot plates on the drying process. The glazed non-porous

surface combined with its curved concave well shape affected the process of evaporation. In

the process of setting, the water evaporated most rapidly generally from the top and shallow perimeter of the well while the thickest center section retained liquid for the longest amount of time resulting in an uneven set. For this reason a second series of shrinkage tests was proposed using porous, flat-bottomed molds (Phase two).
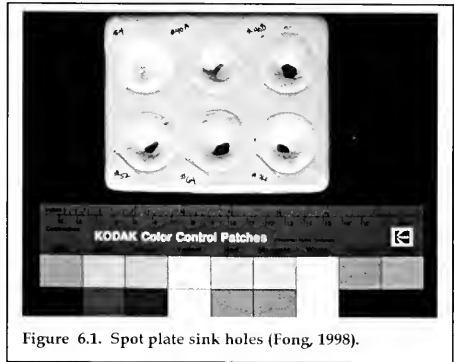

Figure 6.1. Spot plate sink holes (Fong, 1998).

Liquid:Solid Ratios (L:S)

1:1 L:S

The 40% binder concentration category in this grouping displayed unacceptably high levels of air entrainment as did numbers 39, 75 and 87. In conjunction with observations made during the flow test, this category was eliminated from further testing. It has already been noted that the level of air entrainment found within 1:1 L:S was unacceptably high. These formulae were retained from the flow test for this stage of shrinkage test as potentially useful reference points. Numbers 51 and 63 fell within the speculated appropriate M:S ratio range and were promoted to the next phase of shrinkage test.

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[3] The shaded areas in Table 6.2 Shrinkage Phase One Test Sample Matrix represent the combined assessments of the flow and spot plate shrinkage tests.

1:2 L:S

In the 40% concentration category shrinkage failure and air entrainment slightly decreased. Number 65 was eliminated from further testing for the combined factors of excessive air entrainment and weight as observed in the flow test (longest drip of this category - 1:2 L:S, 40%). In the neat concentration (N) viscosity improved and air entrainment again decreased, although not completely. These formulae were free of shrinkage failure and cured the quickest. No other formulae were eliminated from this category.

1:3 L:S

These formulae displayed the least shrinkage of all L:S groupings judging from their dimensional stability. The 40% concentration category exhibited minimal shrinkage. The neat formulations were free from shrinkage failure – their surfaces level and free from cracking - and set the quickest of all categories.

In light of the uneven evaporation as caused by the spot plate mold shape and hydrophobic glazed surface, the neat (N) results reveal that high polymer solids content decreases the rate of shrinkage.

Phase Two – Unglazed Ceramic Dishes[4]

Liquid:Solid (L:S) / Binder Concentrations

	1:1			1:2			1:3			1:4		
M:S	**20%**	**40%**	**N**	**20%**	**40%**	**N**	**20%**	**40%**	**N**	**20%**	**40%**	**N**
1:0	1	2	3	4	5	6	7	8	9	10	11	12
0:1	13	14	15	16	17	18	19	20	21	22	23	24
1:1	25	26	27	28	29	30	31	32	33	34	35	36
2:1	37	38	39	40	**41**	42	**43**	**44**	45	46	47	48
3:1	49	50	**51**	52	**53**	54	**55**	**56**	**57**	58	59	60
3:2	61	62	**63**	64	65	66	**67**	**68**	69	70	71	72
4:1	73	74	75	76	**77**	**78**	**79**	**80**	**81**	82	83	84
5:1	85	86	87	88	**89**	90	**91**	**92**	**93**	94	95	96

Bold numbers indicate formulae tested during this phase.
Shaded areas indicate those formulae deemed unsuccessful.
Table 6.3 Shrinkage Phase Two Test Sample Matrix

General Observations

Small shrinkage cracks were visible in numbers 51 and 63 only. No sinkholes occurred, undoubtedly due to the even absorption and evaporation of the water content. High air entrainment was visible in numbers 51, 63, 53, and 89 (30 – 50 % aeration). Percent solids ratios influenced surface tension visible as contact angles and meniscus curves. Surface tension is related to bulk viscosity, which is a product of particle solids content.[5] Increased solids content produced surface profiles consistent with the initial contact angles in both the wet and dried states of the formulae. The surface profiles of those formulae with the highest concentration of polymer solids (57, 81 and 93) were convex in both the wet and dry states, conversely the surface profiles of the 20% formulae were concave in both the liquid and dry states. The concave surface profiles of numbers 51, 63 and 41 indicated a low solids content

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[4] The shaded areas in Table 6.3 Shrinkage Phase Two Test Sample Matrix represent the combined assessments of the flow, and phase one and two shrinkage tests.

[5] A. N. Gent and G. R. Hamed, "Fundamentals of Adhesion," *Handbook of Adhesives* 3rd ed., Irving Skeist ed., (New York: Van Nostrand Reinhold, 1990), p. 45.

and associated shrinkage. The surface profile appeared even at number 43 in both the wet and dry states. It is worth noting that this was the first formula of the 1:3 L:S grouping tested at this stage, further supporting the speculation that the ideal L:S ratio was 1:3. As the solids content increased so too did the viscosity (as evidenced in section 6.1 Flow) which in turn affected surface tension, evidenced in surface profile and subsequent shrinkage.

Solids and liquid contents are in inverse proportion to one another. A high solids fraction (both polymer and filler) increases viscosity and decreases shrinkage. An increase in viscosity also increases surface tension and lowers wettability of the grout.

Liquid:Solid Ratios (L:S)

L:S 1:1

Formulae 51 and 63 displayed small shrinkage cracks and high degrees of aeration (50%). These two were eliminated from further testing based on the combined results of the flow and shrinkage tests.

L:S 1:2

All mixtures from this grouping were eliminated from further testing due to unacceptable levels of air entrainment observed in both flow and shrinkage tests (30%).

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[5] A. N. Gent and G. R. Hamed, "Fundamentals of Adhesion," *Handbook of Adhesives* 3rd ed., Irving Skeist ed., (New York: Van Nostrand Reinhold, 1990), p. 45.

L:S 1:3

Six mixtures were selected for further testing, numbers 55, 79, 56, 80, 57 and 81. This selection provided a range of comparable mixtures from each binder concentration from the most successful filler ratio combinations. Numbers 91, 92 and 93 were eliminated from further testing for their low sand fraction. Previous research has established that sand is a critical element in imparting strength to filled adhesive systems.[*] Numbers 67 and 68 were eliminated for their high sand fraction which flow tests revealed produced a heavy grout and consequently failed to fulfill the criteria of appropriate strength and light weight.

The collective results of the flow and shrinkage tests indicate that the ideal mixture will be located in the 1:3, L:S grouping at either the 20% or 40% binder concentrations. While the neat category may offer minimum shrinkage, high strength, cohesion and adhesion, the high resin content will result in low (poor) water vapor permeability, a very important property and greater flex that may not be desirable.

[*] Matero and Bass, "Design and Evaluation."

6.3 Dispersion

Dispersion observations were made from cross sections of the unglazed ceramic dish samples.

General Observations

All samples exhibited even dispersion of solids and good adhesion and cohesion. There were no visible voids between the grouts and the ceramic dishes. It is clear that the sand component has a direct affect upon aeration. Its presence inhibits air entrainment. As noted before, the higher the solids content, the lower the shrinkage and degree of aeration.

Liquid:Solid Ratios (L:S)

1:1 L:S

As expected, numbers 51 and 63 experienced the most shrinkage and highest degree of aeration (50%). This was visible in the thinness of the dried grout layer as observed in cross section. These two formulae were eliminated from further testing.

1:2 L:S

Number 41 displayed a distinct decrease in air entrainment (to a final fraction of approximately 30%) from formulae in the 1:1 L:S grouping (average aeration 50%). Air entrainment increased again in number 53 due to the reduction in sand. Air entrainment decreased in number 77 where it was less than number 53 and more than number 41. Number 89 displayed a higher degree of aeration than number 77. These four formulae (41, 53, 77 and 89) clearly exemplified the relationship between filler solids and air entrainment. The effect of

the sand fraction was easily isolated in this instance where the L:S ratio and binder concentrations were constant. The size, weight and presence of the sand inhibited aeration in the grout mixture. This pattern was repeated to a slightly lesser degree in the neat formulae, (54, 78 and 90).

1:3 L:S

All formulae in this grouping exhibited good even texture, minimal aeration and minimal or no shrinkage.

6.4 Shear Strength Under Compressive Force

Assembly Fabrication

Observations

Grout surface tension became a practical issue during this test. Surface tension is an important characteristic of the grout related to viscosity and thixotropy and relevant to the mode of application. A grout lacking thixotropic properties (low viscosity and surface tension) will run and fail to fill the identified gap. A grout with too high a surface tension will experience difficulty flowing into the narrow space of the gap. The flow test tested unrestricted flow however the grout will be constrained by two surfaces of friction. Application method will also influence the design of appropriate surface tension. Full film fills will require a lower surface tension than spot welds. These factors must be taken into consideration when making a final decision on a grout formula. The average amount of grout fill per strength test

assembly was approximately 18 cc. Injection amounts ranged from 30 cc for number 79 to 15

cc for number 81. No single mixture flowed evenly throughout the gap. Every assembly was

topped off with final injections into the visible gaps from the open top edge down. The

viscosity and subsequent surface tension of the grouts was too high to easily and completely

cover a vertically oriented volume of approximately 166 cm^3. The neat formulations (57 and

81) were extremely difficult to pass through the needle thus exhibiting poor injectibility. Their

high resin solids content resulted in an unacceptably high surface tension and poor flow - poor

coverage, and appeared to reduce the working time - quicker set. These obstacles passed

unnoticed in the earlier tests that required small batches for discreet unconstrained

applications. The larger volumes of grout required for this test and the physical constraints of

the test models increased the demand for a flexible working time. In their current state, these

two formulae (57 and 81) are not recommended for the identified application - Type A

reattachment.

Liquid:Solid (L:S) / Binder Concentrations

M:S	1:1			1:2			1:3			1:4		
	20%	40%	N	20%	40%	N	20%	40%	N	20%	40%	N
1:0	1	2	3	4	5	6	7	8	9	10	11	12
0:1	13	14	15	16	17	18	19	20	21	22	23	24
1:1	25	26	27	28	29	30	31	32	33	34	35	36
2:1	37	38	39	40	41	42	43	44	45	46	47	48
3:1	49	50	51	52	53	54	**55**	**56**	**57**	58	59	60
3:2	61	62	63	64	65	66	67	68	69	70	71	72
4:1	73	74	75	76	77	78	**79**	**80**	**81**	82	83	84
5:1	85	86	87	88	89	90	91	92	93	94	95	96

Bold numbers indicate formulae tested during this phase.
Shaded areas indicate those formulae deemed unsuccessful.
Table 6.4 Shear Strength Under Compressive Force

Test Results

The limited amount of available building material yielded three assemblies per formula. This presented distinct limitations on the reliability and accuracy of the data. However, given the limitations, the six tested formulae did produce a pattern of logical results.

> It can generally be stated that there is an inverse relationship between the flexibility and film strength of the adhesive - the higher the strength, the lower the flexibility... Thus most conservation applications benefit from flexible or semi-flexible adhesives.[7]

General Observations

The results of this test confirmed that resin content is the most influential factor in determining strength, and that a higher sand ratio proportionately also contributes to increased strength.[8] The neat formulae exhibited the greatest strength and the higher sand fraction consistently provided the higher strength value for each binder concentration tested. The patterns of failure confirmed theories regarding the effect of solids content of both polymer concentration and filler fraction on,

- cohesion
- adhesion
- surface tension/flow, and
- penetration/wettability.

Inherently, emulsions possess limited pore penetration ability due to their large molecular size. These resin particle droplets are long, large polymer molecules. The *size* of the molecule

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[7] J.L. Down, M.A. MacDonald, J. Tetreault and R.S. Williams "Adhesive Testing at the Canadian Conservation Institute- an Evaluation of Selected Poly(vinyl acetate) and Acrylic Adhesives," *Studies in Conservation* **41** (1996): p. 30.

limits the space through which it can fit or its ability to penetrate another material. The large polymer molecules are dependent upon the water fraction to carry them into the adjacent material, particularly when encountered with material of low porosity such as the caliche. The very small molecule size of water results in excellent penetration ability (high wettability). Increased resin particle concentration/low water content induces higher viscosity. High viscosity produces high surface tension, which reduces flow (coverage). These factors have a direct affect upon adhesive strength where surface contact and penetration are essential in establishing good adhesive bond.

In short,

- high polymer content increases cohesive strength and viscosity (surface tension)

this is turn,

- decreases flow (coverage), penetration/wettability thereby reducing points of contact for adhesive bonding.

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[8] See Matero and Bass, "Design and Evaluation." That the sand ratio has a direct effect upon strength is consistent with the findings of Matero and Bass in their development and evaluation of hydraulic lime grouts.

Binder Concentration (%)

20% - Nos. 55 and 79

Failure within numbers 55 and 79 typically occurred within the grout. The low occurrence of adherend/grout interfacial failure indicated successful preinjection procedure, sufficient penetration, and good adhesive bond strength.


Figure 6.2. Numbers 55 (left) and 79 (right), failure typically intra-grout (Fong, 1998).
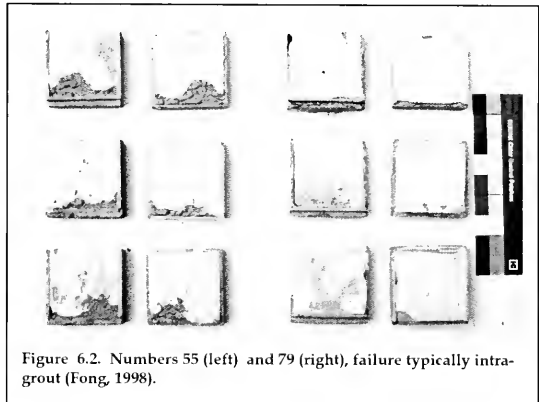
40% - Nos. 56 and 80

Failure within numbers 56 and 80 occurred both within the grout and at the grout/adherend interface. The plane of failure was never continuous within an assembly. Point of failure frequency (intra grout or at interface) was nearly equally divided.

Figure 6.3. Numbers 56 and 80. Failure was fairly evenly distributed between intra-grout and grout/adherend interface (Fong, 1998).
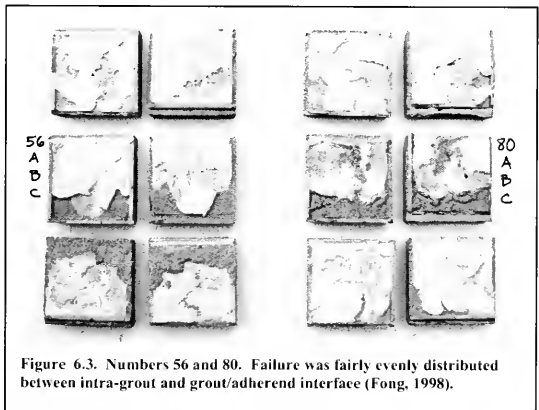
Neat - Nos. 57 and 81

Failure typically occurred within the adherend. Failure within the adherend does not meet the criterion of sufficient strength. In this instance, the cohesive and bond strength of the grout exceeded the cohesive strength of the adherend. Failure within the adherend is not acceptable as the end-goal is the preservation of the original fabric. Excessive strength in an adhesive results in

Figure 6.4. Numbers 57 and 81, failure occurred within the adherend (Fong, 1998).
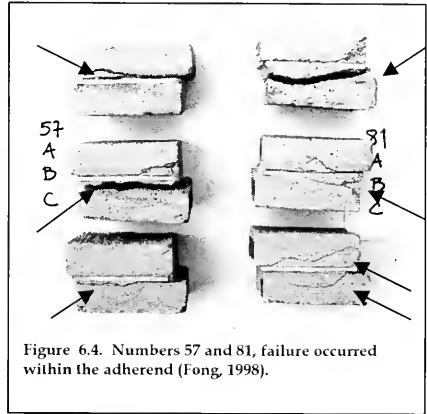
destruction of surrounding fabric as the exerting stress is transferred to the weakest point. Distribution (coverage) of the grout throughout the gap decreased from the 20% and 40% formulae (55, 79, 56 and 80). Incomplete coverage was suspected based on injection difficulty experienced during fabrication. It is useful to note that even with incomplete coverage, formulae nos. 57 and 81 still offered the greatest strength. Formulae nos. 57 and 81 were eliminated for their excessive strength.

SHEAR STRENGTH

ASTM D3931 - Standard Test Method for Determining Strength of Gap-filling Adhesive Bonds in Shear by Compression Loading[1]

	55A	55B	55C	79A	79B	79C	56A	56B	56C	80A	80B	80C	57A	57B	57C	81A	81B	81C
Load (lb.)	140	280	270	360	180*	190	340	245	280	240	150	365	440	330	285	310	275	225
Stress (psi) at point of failure	45. 02	90. 03	86. 82	115. 76	57. 88	61. 09	109. 32	78. 78	90. 03	77. 17	48. 23	117. 36	141. 48	106. 11	91. 64	99. 68	88. 42	72. 35
Average Stress (psi)†		-88.43			-59.49			-84.40			-62.70			-98.88			-94.05	

*Test was stopped and restarted once already begun in order to readjust placement of assembly within the machine. Shaded data are those figures rejected for their degree of difference amongst the three data points. Averages were calculated according to the two most similar data points.

†All numbers in table are actually negative. This indicates *compressive force*.[2]

Table 6.5 Shear Strength Data

Equipment: 19 assemblies, 3 per formula, 1 for initial machine calibration

Instron Testing Machine Model 1125

cross head speed = 0.1 inch per minute

chart speed = 5 inches per minute

Assorted shims

Calculations

Stress (psi) = Load (lb.) / Cross section area

Where cross section area =   3.11inches² (typ.)
                             2016mm² (typ.)

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[1] "D3931, Standard Test Method for Determining Strength of Gap-filling Adhesive Bonds in Shear by Compression Loading," (Philadelphia: ASTM, 1990). Modified by author.

[2] As per Dr. Alex Radin at the Materials Testing Laboratory of the Laboratory for Research on the Structure of Matter (LRSM).

6.5 Water Vapor Transmission (WVT)

The water vapor transmission and strength tests were run concurrently therefore elimination of formulae from one test did not affect their inclusion in the other test.

General Observations

As expected, the polymer solids content was the most influential factor in affecting the water vapor transmission rate. The higher the polymer concentration, the lower the WVT rate. There was no discernible pattern in the effect of the filler ratios (M:S). Perhaps the tested ratios were similar enough to offer little distinction.

Binder Concentration (%)[9]

20% - Nos. 55 and 79

This category had the highest average WVT rate.

No. 55   4.96 g/h·m^2
No. 79   4.82 g/h·m^2

AVERAGE WATER VAPOR TRANSMISSION RATE

Table 6.6

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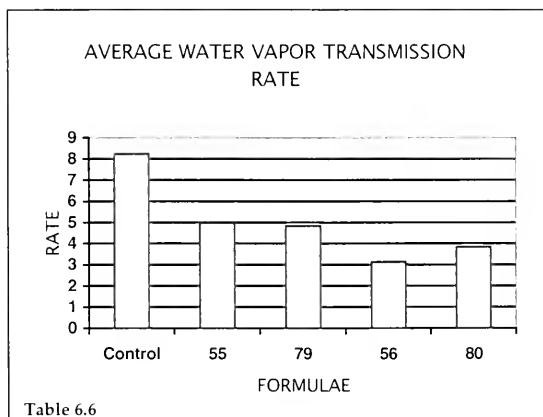
[9] The control sample was untreated earth, (without a grout film).

40% - Nos. 56 and 80

As anticipated, the WVT rate decreased with the increase in polymer solids content.

    No. 56   3.12 g/h·m^2
    No. 80   3.83 g/h·m^2

Neat - Nos. 57 and 81

The data in this category did not follow the same pattern of progressive weight loss observed in the 20% and 40% categories. For reasons not yet clear, the neat formulations occasionally gained weight. There was no discernible pattern to the weight gain. Due to these as yet inexplicable consistencies, the neat formulations were excluded from further calculations. However, available data revealed rates of 0 at T6 for both numbers 57 and 81 and 0.57 at T7. These rates are unacceptably low. The high resin solids content effectively created a near impermeable barrier. The low WVT rate failed to fulfill the criterion of water vapor permeability. An impermeable barrier is materially and functionally incompatible with the original earthen fabric. This barrier would trap internal moisture within the substrate creating adverse environmental conditions for the building material. Water is the most powerful deteriorating force upon earthen material. It induces both physical and chemical effects. Trapped moisture accelerates the natural rate of decay. It is essential that proposed conservation solutions do not create nor contribute to accelerated decay of the structure. Based on the results of this test, neat formulae numbers 57 and 81 were eliminated.

Calculations

Water vapor transmission rates were calculated according to the following methodology and equations.

According to guidelines provided by both ASTM and NORMAL, calculations for WVT are based on figures from the steady state. The steady state is that period of time in the test where weight loss between intervals reaches its most consistent level. This point is identified by plotting $\Delta M / \Delta t$. At least two points are necessary from the steady state in order to calculate WVT.

Where M = average weight difference of formula n between each interval
    t = time in hours of each interval
    Tn = point (date and time) of weighing

ex.) no. 55

    ΔM at T6 = weight of sample at T6 from weight of sample at T5
                      The average weight of 55 A, B and C at T6 is 0.44g

    Δt at T6 = time interval from last weighing
                      The time interval from T5 to T6 is 23.95 hours

Therefore,

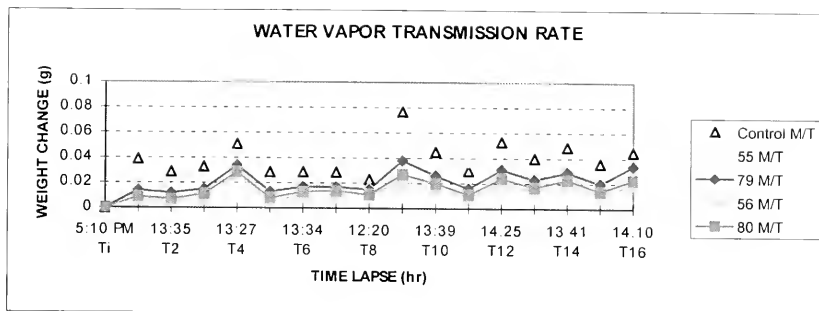 $\Delta M / \Delta t$ = 0.44 g / 23.95 h = 0.018 g/h

Table 6.7

Table 6.7 reveals that the steady state exists between T6 and T7. The fluctuations in weight loss that occur after the steady state are attributable to desiccant change. In an effort to control the RH within the chamber the desiccant was changed at T9, T12, T14 and T16. However, the effect of this action was a seemingly erratic sequence of difference in ΔM / Δt.

WVT is defined as $(\Delta M / \Delta t) / A$

Where A = surface area (m²) of disk
        A = .003527 m²

ex.) no. 55

        WVT at T6 =
        0.018 / .003527 m² = 5.103 g/h·m²

101

WATER VAPOR TRANSMISSION
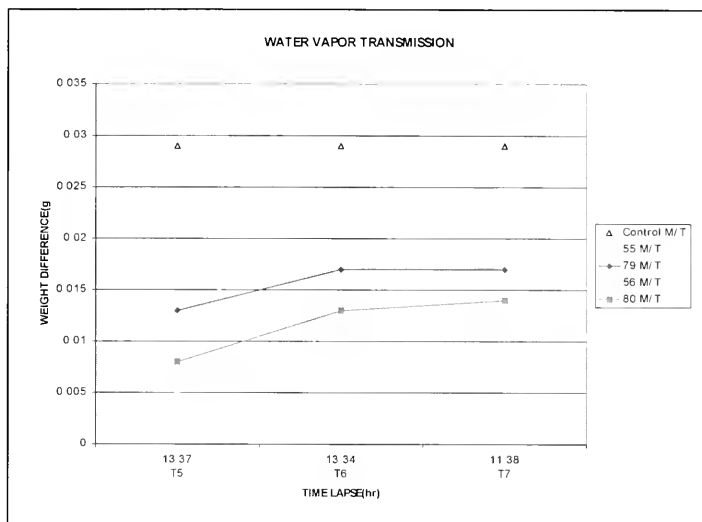
Table 6.8

Margin of Error

All mathematical figures offered in this section are relative, none are absolutes. The selection of data used for calculations is rooted in standard theory and methodology however, the uncontrollable variables of the test such as fluctuating temperature and RH within the desiccation chamber, and the imprecision of some of the measuring devices such as the hygrometer, do not produce consistent incontrovertible data.

Conclusion

The relative performance results were entirely consistent and useful. All resultant data was relative to the control (untreated) sample and the percent solids of resin. This test revealed that of the six formulae tested numbers 55 and 79 had the highest WVT rate and are therefore the most desirable. However, 'high' is a relative term. The WVT must be similar enough to the original material to be viewed as an acceptable treatment option. The viability of this option will also depend upon method of application (i.e. full film or spot weld).

The difference in WVT rates between numbers 55 and 79, and numbers 56 and 80 were significant. Number 55 produced a relative WVT rate $22^o/o$ higher than that of number 56 and number 79 produced a relative rate $12^o/o$ higher than that of number 80. The higher WVT rates of numbers 55 and 79 ($20^o/o$ binder concentrations) can be attributed to their reduced polymer solids contents. The test also revealed that within the steady state, the difference in WVT rates between numbers 55 and 79 was so slight as to be negligible (less than $2^o/o$ with number 55 producing the higher rate). This indicates that all other cohorts being consistent, (binder concentration - $^o/o$, L:S ratio, amount used and application method) the difference between a 3:1 and a 4:1 M:S ratio upon WVT is minimal.

This outcome was reversed between numbers 56 and 80 where number 80 produced a relative WVT rate approximately $8.5^o/o$ higher than number 56. Is there a relationship between polymer resin content and filler ratio which can reverse results of WVT rates from formulae of identical proportions but differing resin contents, or is this just an anomaly due to an inconsistency in the test? Further testing and research could answer these questions.

6.6 Laboratory Conclusions

Of the 100 grout formulations tested number 55 performed best in each property assessment for flow, shrinkage, strength, dispersion and water vapor transmission. Formula number 79 exhibited a comparable water vapor transmission rate to that of number 55 however its strength performance was much lower than number 55. For this reason, number 55 was selected for final trial field tests. The issue of treatment compatibility remains depending upon what are acceptable values or limits for each property. Flow, shrinkage and dispersion are properties that can be internally assessed relative to the other grout formulations. Strength and water vapor transmission must be assessed relative to the whole system (earthen materials, detachment gaps, and method of application).

- How much strength is necessary?
- How porous are the adherends?
- What are the ambient weather conditions of the site, (temperature and moisture cycles)?

The answers to these questions will influence the final assessment of the grout performance. It is clear from the laboratory testing programme that number 55 produced the best relative performance results given the identified criteria. However, relative and acceptable are two distinctly different assessments. When possible, it is always desirable to implement a pilot field test to more fully assess a treatment's actual performance. The uncontrollable features of a site and the passage of time can never fully nor accurately be replicated in a laboratory environment, although they can be artificially simulated to a certain degree.

7.0 FIELD TESTS

This section offers very brief descriptive, qualitative assessments of grout number 55 trial tests at four different field sites. These trial tests were conducted during the summer of 1998. These four sites were:

- The Solitude, Philadelphia, Pennsylvania

- Kiva Q, Cliff Palace, Mesa Verde National Park, Mesa Verde, Colorado

- Historic Building 15, Fort Davis National Historic Site, Fort Davis, Texas, and

- The Casa Grande, Casa Grande Ruins National Monument, Coolidge, Arizona.

Not all of these tests were performed by the author and some of the following information was gathered from conversations between the author and those conservators who performed the treatment applications.

The Solitude, Philadelphia, Pennsylvania- Late 18th century residence of Thomas Penn

The grout was used for an interior application at the wall and ceiling juncture. It was injected between the wood substrate wall and a plaster molding. The grout was injected behind the molding according to standard procedure. The grout was administered June 1998. As of September of the same year it was deemed successful in application and as cured treatment.

Cliff Palace, Mesa Verde National Park, Mesa Verde, Colorado – 13th century alcove dwellings

The grout was used for an exterior application. It was injected behind layers of thin, highly absorbent earthen plasters. The plasters immediately absorbed the liquid emulsion. This produced two undesirable results; one - the solids fraction remained as an ineffectual

segregated component, two - the emulsion stained (darkened) the plasters. The grout was deemed an inappropriate treatment for the thin finish (wash) context.

Historic Building 15, Fort Davis National Historic Site, Fort Davis, Texas –
Mid 19th century officer's quarters

The grout was used for an unprotected exterior application. It was injected behind thin layers of lime plaster against an intermediary lime scratch coat. The treatment was used throughout the structure. It was found to be useful in areas too thin/fine for the lime grout. Grout was recessed from the exposed plaster edge by approximately 0.6 cm and capped with a fine edging material of fine silica sand, hydraulic lime and water. It was during these field trials that the grout viscosity was found to be respondent to heat.

The Casa Grande, Casa Grande Ruins National Monument, Coolidge, Arizona –
Prehistoric (possibly 13th – 14th century) freestanding four storey structure

The grout was used in a sheltered exterior application. A small area in Tier B, room space 2 was selected for a trial treatment. This involved the cleaning and reattachment of a detached earthen layer of approximately 0.16 - 0.6 cm thickness and 20 cm^2 (sub-circular shape). The treatment attempt failed. Failure may be attributed to a number of reasons, individual or combined. The degree of deformation (planar discontinuity) of the detached fragment was pronounced and the surrounding area suffered from cracking. These two factors presented a fragment in a highly precarious state. It is possible that the detached layer was over-saturated with moisture from both the flushing and prewetting preparatory procedures. Over-saturation of earthen material can easily result in loss of internal cohesion and excessive

weight. The facing was not sufficiently adhered to support the combination of the now moisture saturated plaster and the additional stress presented by the grout. Consequently, the fragment collapsed.

8.0 CONCLUSIONS

The Casa Grande appears to be of relatively stable condition. The rate of deterioration has been greatly mitigated since the initial stabilisation efforts of Cosmos Mindeleff and the erection of the protective shelter (1902, 1932). The policy of minimum intervention for the site, adopted at the turn of the 19th century has resulted in a structure of exceptional integrity. Climate, building material and original construction techniques are the primary reasons for the monument's impressive longevity. Policy and restraint are the reasons for its largely unaltered current condition and degree of integrity. Unless the state of deterioration is active and poses an eminent threat to the physical integrity of the Casa Grande, a continued conservative approach to its preservation is strongly advocated. It is this approach which today presents us with such an exceptional example of the past.

Since 1697 when Captain Juan Mateo Manje compared the interior plastered walls of the Casa Grande to polished Puebla pottery these earthen plaster finishes have been recognised for their outstanding quality, condition and extent. They offer a unique glimpse (it is the only example of its type in the region - free standing, monumental plastered caliche construction) into the built world of the region's early inhabitants. The plasters exist in a relatively stable condition in great part due to the protection afforded by the shelter.

The phenomenon of loss is uniform in its effect but multifarious in its causes. Initially distinct and simple causes (mechanisms of decay) are at second glance mere components of a cycle of interdependent and overlapping relationships. Loss attributed to mechanical damage such as

the removal of vigas and lintels creates the conditions for environmental exposure, and animal and insect intrusion. Insect and animal activity, wind, rain and active running water all have physical as well as chemical effects. Any one of these factors can serve as catalyst for a preexisting condition or exacerbate a point of weakness.

This research addressed the issues of plaster finish stabilisation. The process began with reference to historical records of the structure and its condition over time. This was followed by a condition survey and assessment, sample taking, material characterisation and the design and development of a treatment option. Historical records depict a Casa Grande much the same as the one perceived today. The survey and assessment qualified the *in situ* condition of the plasters and identified the endemic condition of Type A (large scale) detachment as well as the less common Type B (small scale/flaking). The extent and unusual nature of Type A presented an unfamiliar condition and consequently posed a potential threat to the integrity and stability of the plasters. Proposals for the mitigation of Type A detachment became the focus of the research.

Type A large scale detachment is typified by the expanse, extent and regularity of the detachment gap 3 mm average, and the evenness of the adherend surface layers. Detached layer thickness is variable 0.16 - 0.6 cm for finish to 0.6 - 10 cm for intermediary. The pervasiveness, regularity, expanse and even surfaces of this condition strongly suggest an intrinsic relationship to original wall construction. This phenomenon was observed during field surveys and documentation and has been described as a system of vertical planar wall layers; generally ascribed the puddled earth substrate, intermediary leveling layers, finish

layer and color wash. The layer construction is evident due to loss, the occurrence of which has created and exposed broken edges, cross sections into the wall itself. These now open and exposed areas produce 'breaks' in the wall as a holistic unit. The damaged areas present points of weakness to the mechanisms of decay (wind, water, animal activity, gravity, debris accumulation, movement) and render them vulnerable to accelerated deterioration. If Type A detachment is a result of construction technique, then it is an inherent and original condition and therefore inactive and stable. However, loss initially caused by extrinsic factors, can exploit preexisting inactive (intrinsic) conditions to produce scenarios of instability.

Research into existing methods of grouting for plaster reattachment expanded to include adhesive, and adobe and concrete amendment studies. The most recent successful grouting method presented by Matero and Bass of hydraulic lime grout proved ineffective for gaps of 3 mm or less. The grout possesses a particle size range and viscosity that prohibit it from flowing into gaps of this size. At the moment there is no clear alternative to the proposed filled grout (no. 55) although it is now clear that its appropriateness is dependent upon the detached layer material, absorbency and thickness.

The earthen nature of the original building fabric informed the design of the grout. A lightweight, non-reactive, aqueous based grout was sought as a compatible treatment alternative. The laboratory testing programme revealed the following corollaries as they pertain to the tested grout formulae,

- sand is a necessary component for strength
- polymer solids content affects water vapor permeability, strength and viscosity

- solids content and shrinkage are in inverse proportion to one another, and
- the sand component must be less than 2/5 and greater than 1/6 of the total filler content to fulfill the criteria of appropriate strength, flow and minimum weight.

Formulae numbers 55 and 79 produced the most desirable results of all 100 tested formulae. Number 55 was chosen for final selection for its greater strength over the incrementally lower WVT rate of number 79.

Pilot field tests of the grout produced informative results. The grout has so far performed successfully for interior applications of lime plaster to wood and exterior applications of thin lime plaster to intermediary scratch lime plaster. It was deemed inappropriate for use with highly absorbent thin layers of earthen washes. The acrylic emulsion resulted in staining of the thin, highly absorbent earthen plasters of Mesa Verde. The results of these field tests conclude that the grout

- is useful for the reattachment of fine finish lime plasters to adobe
- is useful for lime plasters to wood
- is inappropriate for highly absorbent thin earthen plasters (≤ 0.32 cm = 1/8 inch).

However, it may prove useful for circumstances characteristic of the Type A condition where the earthen finish plaster is > 0.32 cm depending upon the level of plaster absorbency.

9.0 RECOMMENDATIONS

The aim of conservation is to manage the inevitable forces of change in order to extend the life of the object or site for the preservation of the cultural and material information it retains and offers. This information is found within the material of the thing itself and the immaterial of that which it signifies. Together these constitute the life of the thing in an interdependent relationship comprised of the physical object/site and the forces that have shaped its existence. Failure to address both aspects results in an incomplete comprehension of its condition and relevance which are both essential, intrinsic components to the development of any treatment programme. Conservation practically aims to mitigate the processes of decay. These efforts are often most skillfully and elegantly realised through the simplest and most unintrusive methods. This is expressed in the philosophical and theoretical criteria of minimum intervention towards the maximum retention of original fabric. This notion implies not only retention of fabric and form but also of inherent characteristics. It encompasses the goals of compatibility (physico-chemical and mechanical/functional), appropriate intervention (material and contextual) and the highly idealised reversibility. Reattachment through rehydration of clays as is proposed for Type B detachment is exemplar of this approach.

The methods and techniques of conservation are specialised and should be administered by trained conservation professionals.

9.1 Specific Treatment Recommendations

Type A (large scale) Detachment

Type A detachment requires a grout with gap filling properties. Injection grouting is the advocated material and method in areas of instability only. Areas of instability are easily identified by bulges or planar discontinuities in the finish layer caused by excessive erosion, loss or the accumulation of debris; cracks; and/or continual direct exposure to extrinsic mechanisms of decay. In these instances, localised, full film grouting is recommended. Full film grouting is not recommended for large expanses. It is not advisable to interject a foreign film between two like materials where none previously existed - even though the film possesses a degree of water vapor permeability - especially where it is not necessary. Until evidence proves otherwise, the condition of the plasters has been identified as generally stable. Treatment should only be administered in those localised areas of instability.

Areas of potential instability should be monitored for change and if possible, the causes of decay alleviated (the purpose of the shelter). Spot welding could be advocated as a preventative measure in areas lacking sufficient structural support. This will require a slight elevation in grout viscosity. This could be achieved by the following means,

- slight increase in filler ratio
- slight increase in binder concentration, or
- slight increase in thickener (concentration or ratio).

The application of treatment (injection grouting) should follow standard protocol as outlined below.

- Survey and assessment
- Documentation
- Mechanical cleaning
- Flushing/prewetting with water:ethanol (1:1)
- Preinjection with 10°₀ E-330 in water
- Injection grouting
- Capping where necessary (exposed edges)

Type B (small scale/flaking) Detachment

Rehydration with a 1:1 water:ethanol mix, administered through spray and/or injection with the aid of cosmetic sponges and/or wet strength tissue. This has already proven successful at the Casa Grande in Tier D, north wall (Matero, 1997).[1]

9.2 Future Research

No research program can address and investigate all of the relevant questions. Instead each program defines its scope and attempts to respond to a series of identified unknowns. In the process of investigation and discovery, invariably unexpected issues are encountered which

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[1] Frank Matero, personal communication with author, 1998

raise new questions. Recommendations for future research include the following research questions,

- What effects, both short and long term, does thickener have upon the grout stability, strength and water vapor permeability?

- How does the grout perform over time? This requires return visits to the original trial field treatments.

- What can other instrumental analyses reveal about

    - the finish plaster

    - the grout

    - the plaster/grout interface?

- How can the working time be extended/made more flexible?

- Is the darkened zone a transformation of applied high heat or absorption of fine surface particulates from burning? Subject samples to fire.

- Is it possible to develop an inorganic grout that works with the inherent properties of calcium carbonate?

10.0 APPENDICES

10.1 Sample Location Map

Sample locations are mapped on the following elevations. These represent the twenty-four samples selected for cross section analysis. The sample identification numbers reflect the original sample numbering system of the original forty samples (see Table 2.1 Bulk Analysis Sample Characterisation for sample descriptions).
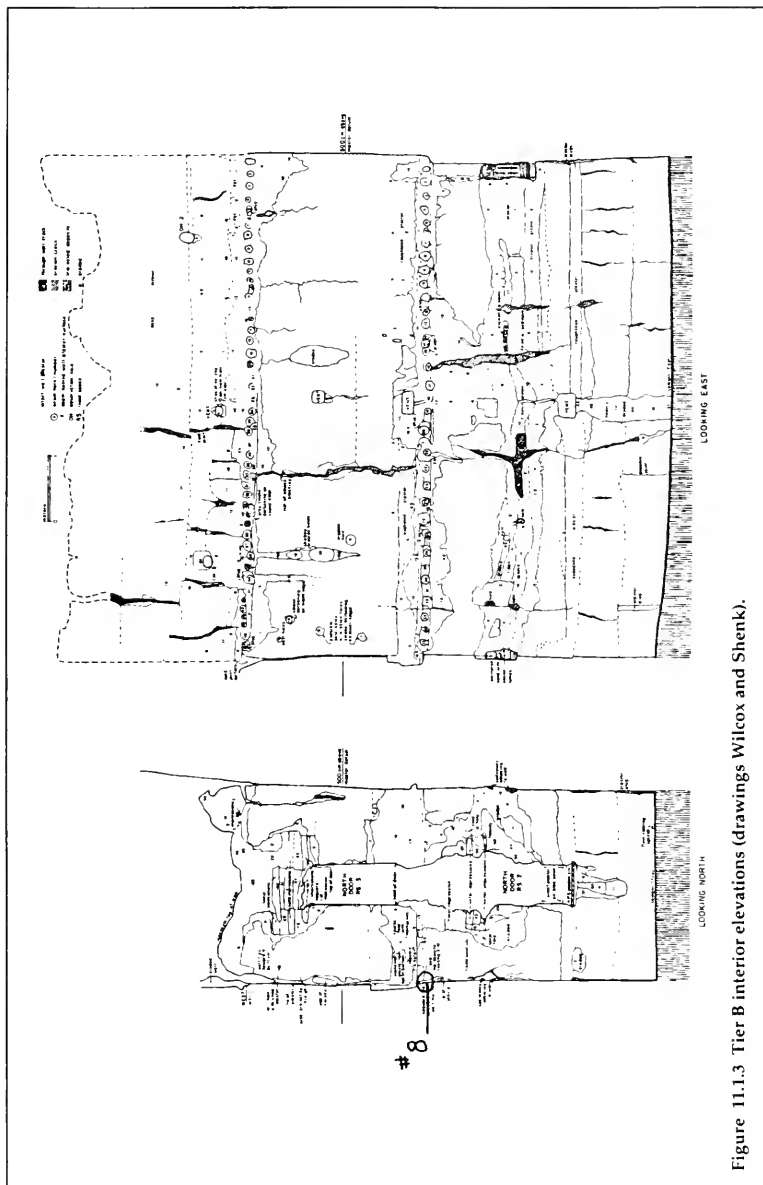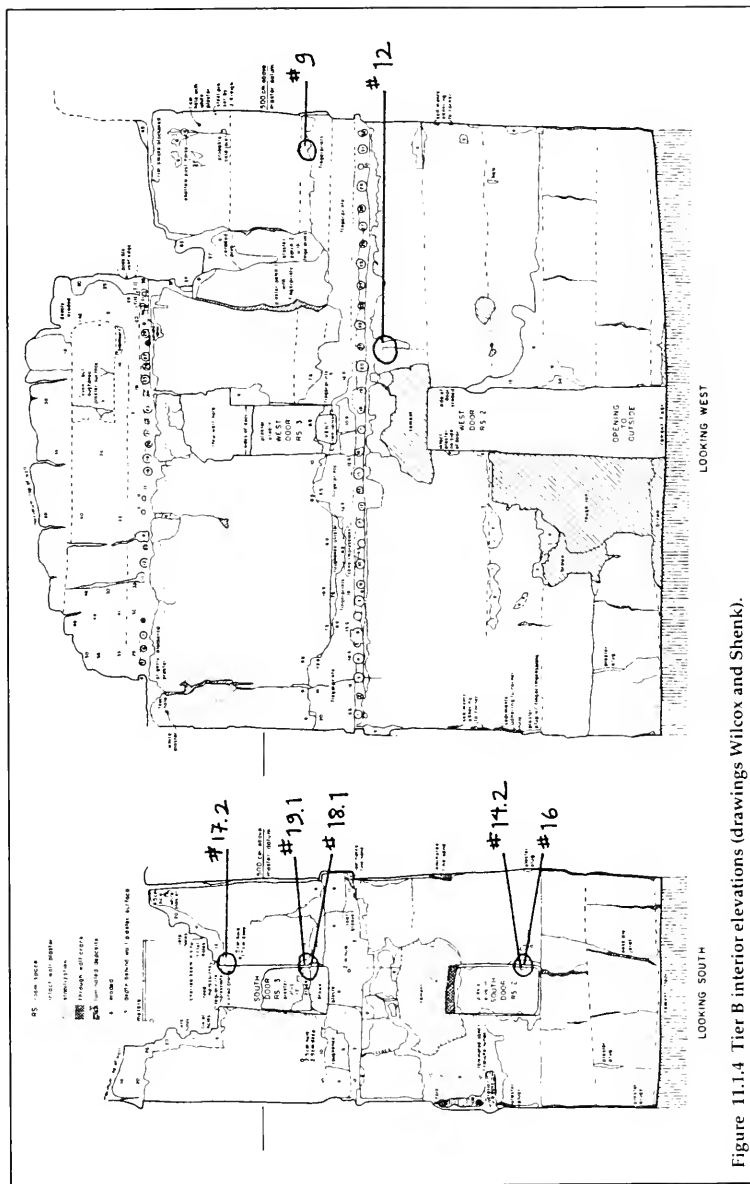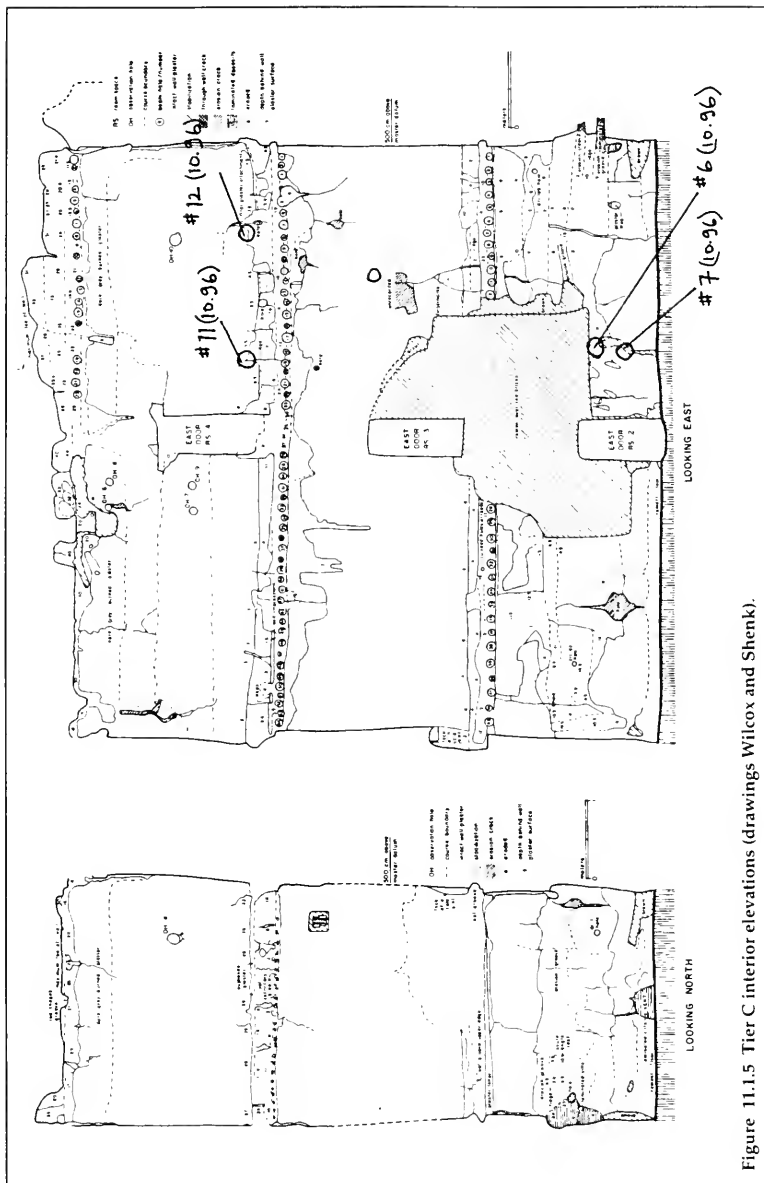
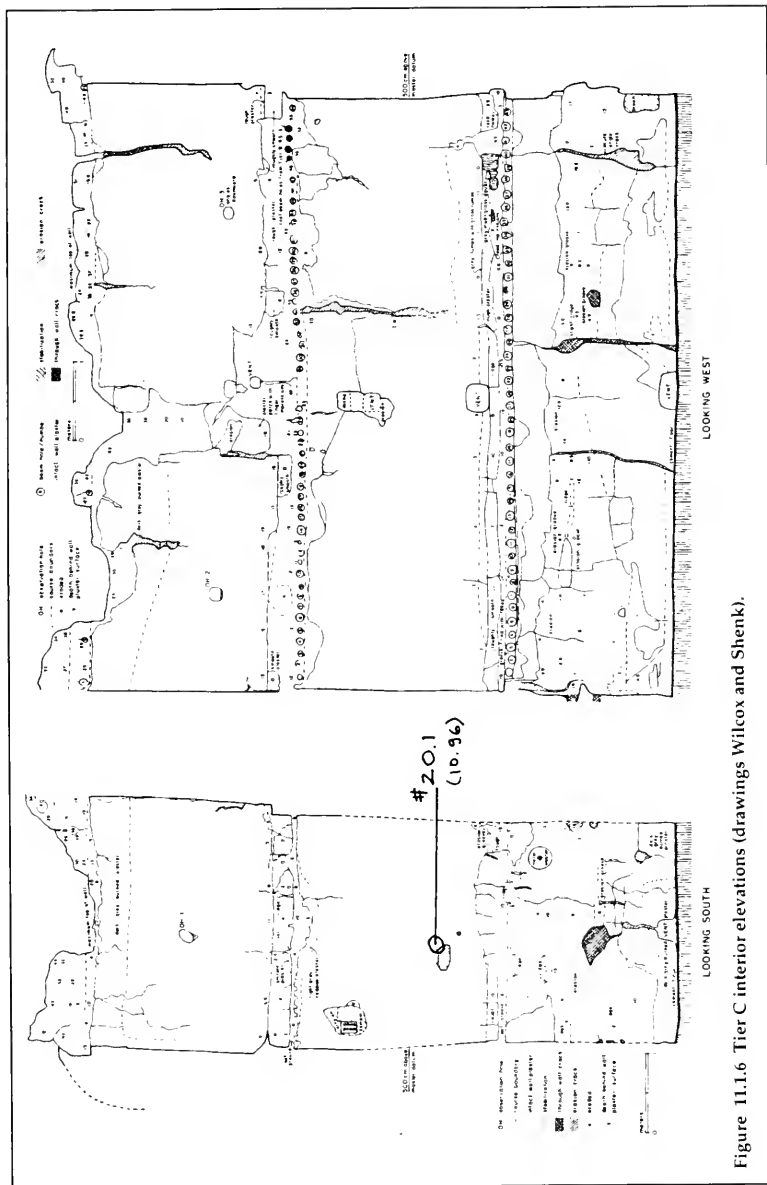Figure 11.1.1 Tier A interior elevations (drawings Wilcox and Shenk).

118

Figure 11.1.2 Tier A interior elevations (drawings Wilcox and Shenk).
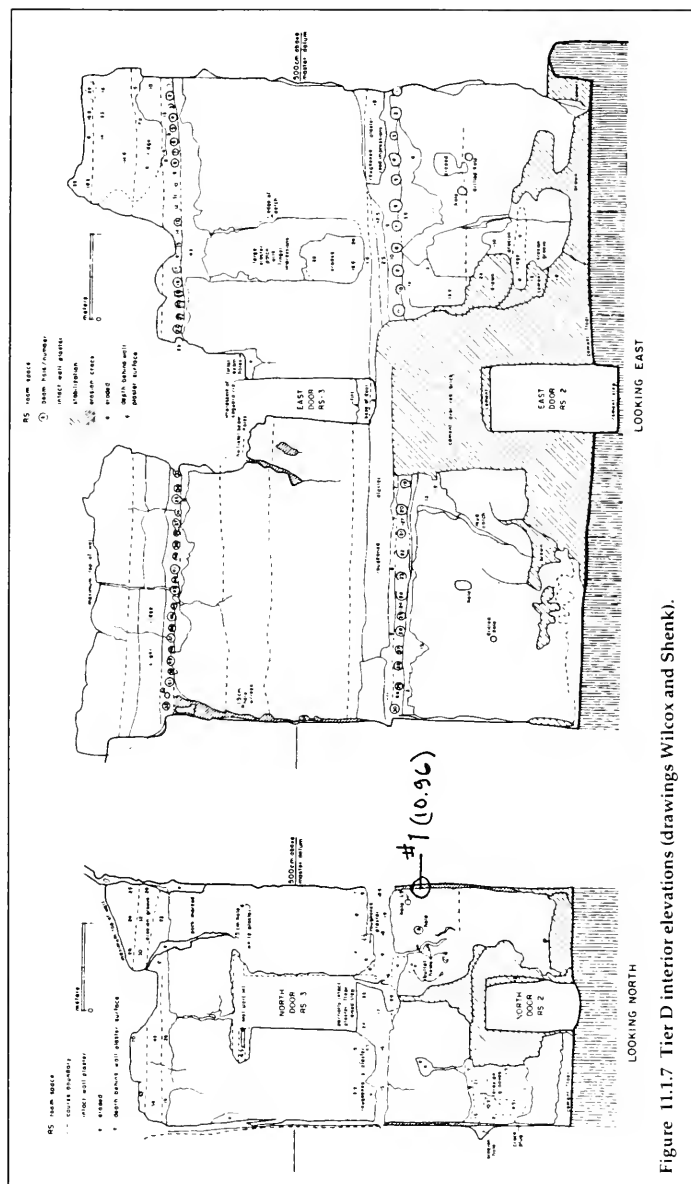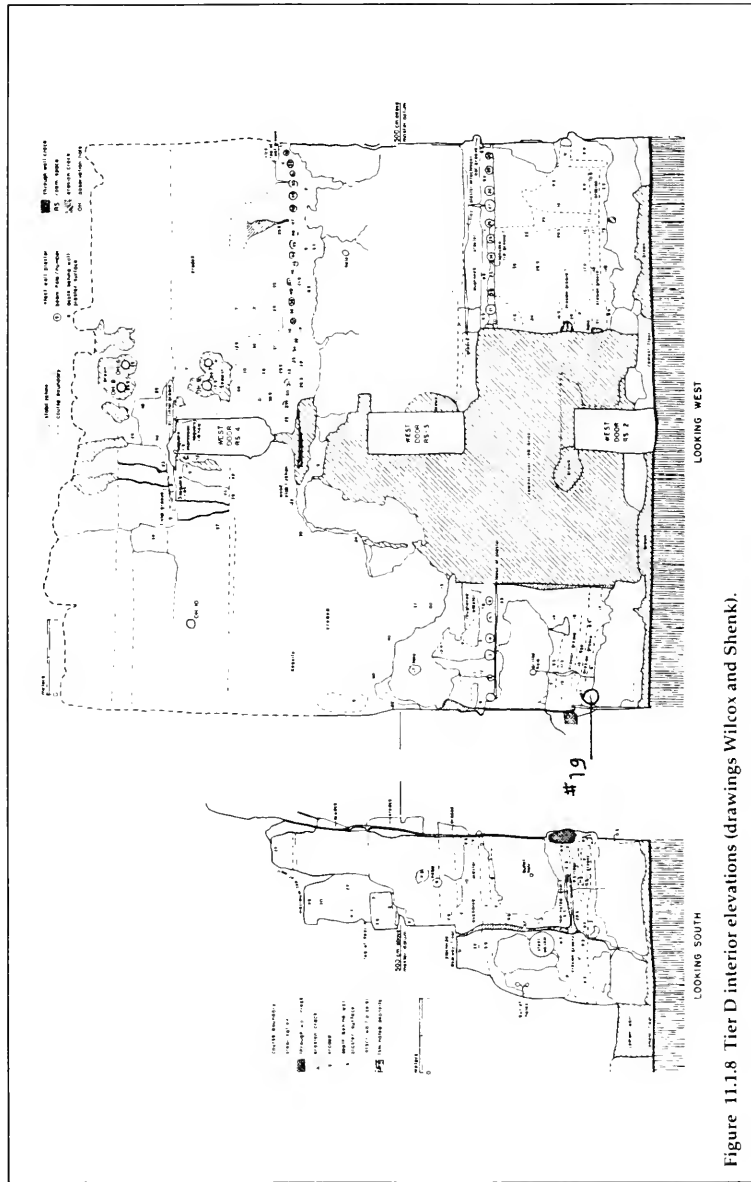
119
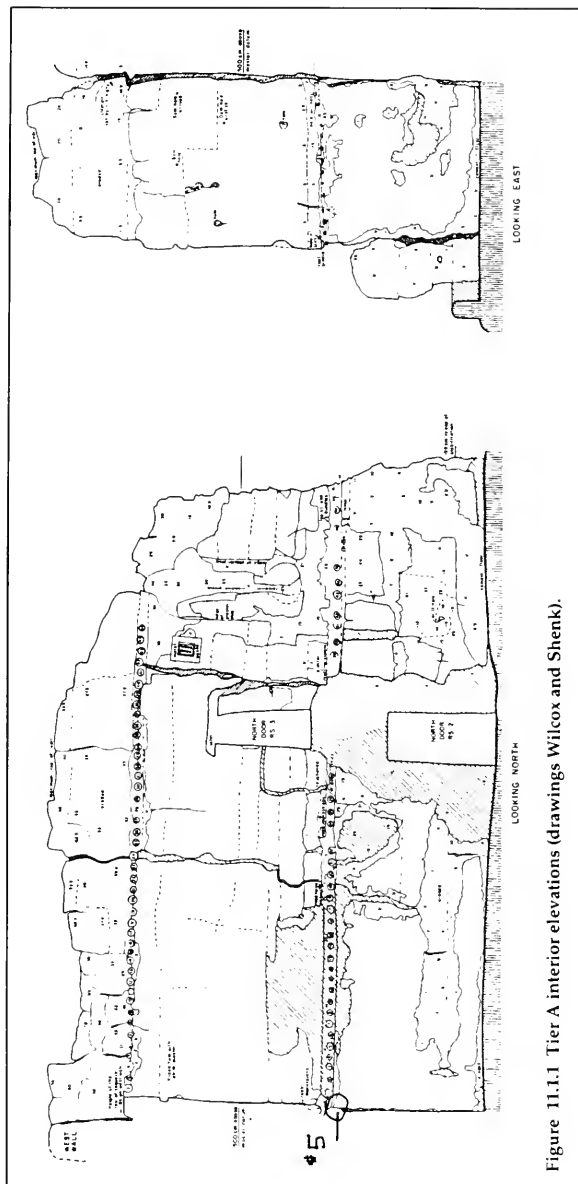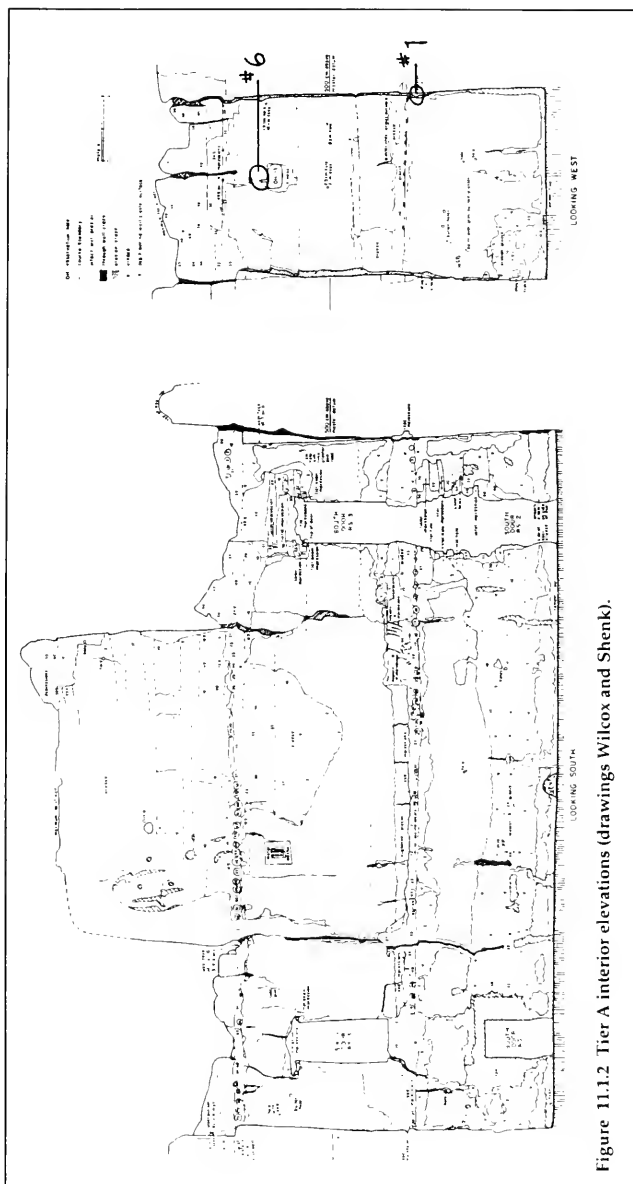
Figure 11.1.3 Tier B interior elevations (drawings Wilcox and Shenk).

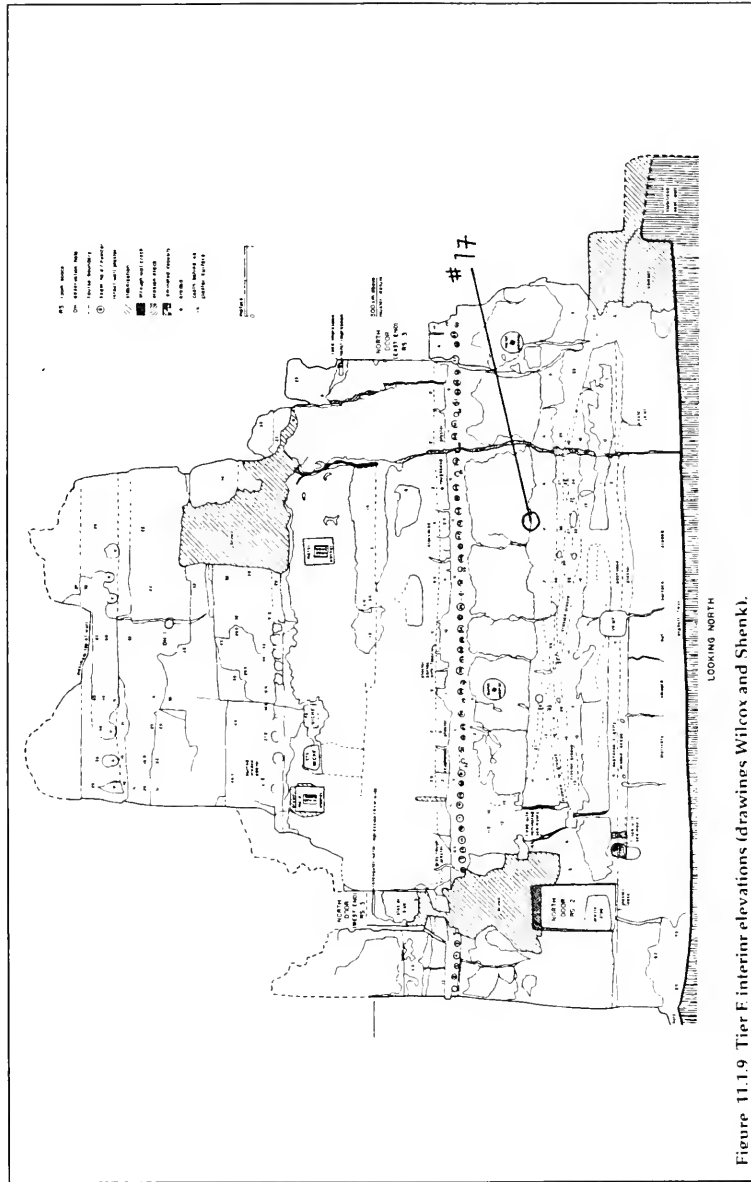Figure 11.1.4 Tier B interior elevations (drawings Wilcox and Shenk).

121

Figure 11.1.5 Tier C interior elevations (drawings Wilcox and Shenk).

122

Figure 11.1.6 Tier C interior elevations (drawings Wilcox and Shenk).

123

Figure 11.1.7 Tier D interior elevations (drawings Wilcox and Shenk).

124

Figure 11.1.8 Tier D interior elevations (drawings Wilcox and Shenk).

125

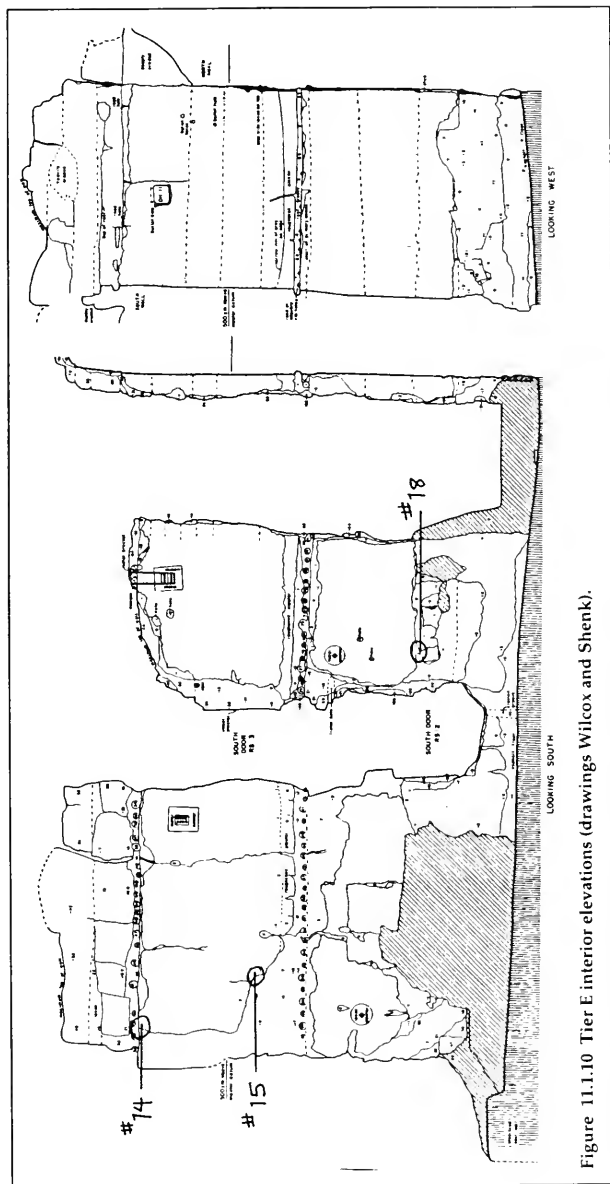Figure 11.1.9 Tier F interior elevations (drawings Wilcox and Shenk).

126

Figure 11.1.10 Tier E interior elevations (drawings Wilcox and Shenk).

127

10.2 Literature Review of Polyvinyl Acetate and Acrylic Emulsion in Conservation

Polyvinyl Acetate (pva) and Acrylic Emulsion (ae); A History of Use in Conservation

Adhesive	Use	Concentration	Location	User/Source	Comments
pva	Fixative for the transfer of Oriental wall paintings	15-30% (15-30 g in 100 cc of solution)		Gettens 1929[1]	
Rhoplex E-330 (ae)	Adobe amendment as consolidant exterior environment	12% 16%	MASCA, UPenn Museum; Hasanlu Tepe, Iran; Chimaltenango, Guatemala; Boca Raton, Florida	Butterbaugh and Pigott, MASCA Mudbrick" 1973-1980[2]	Report filed 1980, tests conducted since 1973. Florida - E-330 12% in sandy clay bricks, after 5 years of weathering, no significant erosion. Supported considerable mildew, which was easily removed with a Clorox solution without damage to the E-330 binding action.
Primal AC-33 (ae)	(preconsolidate d) Mud plaster reattachment	10% in H_2O	Peru	Chiari, Treatment of Adobe Friezes in Peru" 1980[3]	Injection. Part of a three-step preservation process, consolidation, reattachment and paint consolidation and reattachment all *in situ*. Five years later treatment was viewed as successful. This program of treatment was based on the conservator's earlier model at Garagay, Peru 1975-1977.
Primal AC-33 (ae)	Earthen plaster consolidation	5% in H_2O	Samples from Lowry Ruin, CO and LA 17360, NM	Silver, "Architectural Finishes - Prehistoric Southwest," 1980[4]	Brush application - poor penetration and unacceptable change in optical properties.
Rhoplex MC-76 (ae) 47% solids Rhoplex LC-67 Acrysol ASE-60 (thickener) Cabosil M-5 (thickener)	Lime plaster reattachment	Neat (combinations of MC-76 and LC-67 depending on desired strength and flexibility), neat with filler, 3:1/4, 3:1/2 (ae:H_2O) ppv		Phillips, "Adhesives for Reattachment," 1980[5]	Prewetting with 3:3:2 (H_2O:denat. ethyl OH:Rhoplex MC-76)

[1] Rutherford Gettens, "Polymerized Vinyl Acetate and Related Compounds in the Restoration of Objects of Art," *Technical Studies in the Field of the Fine Arts* IV (1935). 15-28.

[2] D. J. Butterbaugh and V. C. Pigott , "MASCA Mudbrick/ Adobe Conservation Interim Report," *Third International Symposium on Mudbrick (adobe) Preservation* (Ankara ICOM & ICOMOS, 1980), p. 19-29.

[3] Giacomo Chiari, "Treatment of Adobe Friezes in Peru," *Third International Symposium on Mudbrick (adobe) Preservation*, p. 39-47.

[4] Constance S. Silver, "Architectural Finishes of the Prehistoric Southwest: A Study of the Cultural Resource and Prospects for its Conservation," 1987, unpublished report. Philadelphia, Architectural Conservation Laboratory, University of Pennsylvania. p. 147. Tests here cited were conducted in 1980.

Adhesive	Use	Concentration	Location	User/Source	Comments
Hydrated lime, microballoons IG-101, fluid coke					
CM Bond M-1 pva	Earthen plaster reattachment	25% solids, 20% dilution[6]	Kiva C, Mug House, Mesa Verde	Silver, "Architectural Finishes," 1981[7]	Prewet with H_2O. Emulsion was injected intralayer, gentle pressure applied to surface with plywood press through wet strength tissue. Silver deems successful.
pva , Primal AC-33 (ae)	Reattachment of lime plaster	pva - 10%, 30-60% with H_2O; ae - 20% in H_2O	Goreme Valley, Turkey	Schwartzbaum, "The ICCROM Project for Conservation," 1971-1983[8]	Prewetting with H_2O and OH
Primal AC-33 (ae), Curasol (pva), Mowilith DM1 H (pva)	Surface consolidation of earthen plasters	ae - 4% in distilled H_2O; Curasol pva - 4% in distilled H_2O; Mowilith DM1 H pva in distilled H_2O 4%	Coastal archaeological sites, Peru	Argumedo "Mural Painting on Adobe Walls," c.1986[9]	ae injection application unacceptable results, darkened surfaces to a dark brown. pva (Curasol) injection application good results in coastal areas but poor in Andean (mountainous and dry) regions. pva (Mowilith DM1 H) injection application good consolidation but darkened surfaces This article also cites another project where a paste was made with a 10% solution of Mowilith, fine sand, and lime for plaster edge consolidation - no proportions given.
Rhoplex MC-76 (ae) Rhoplex LC-67 (pva)	Reattachment of lime plaster via injection	neat		Phillips, "Experiences in the Use of Acrylic Plaster Adhesives,"	Phillips states, "In the publication of 1980, pyrogenic silica (fumed silica) was mentioned as an alternative thickener for the filled

[5] Morgan Phillips, "Adhesives for the Reattachment of Loose Plaster," *APT Bulletin* **12** (1980): 37-63.

[6] Constance S. Silver, "The Conservation of Tempera Mural Paintings and Architectural Finishes," *Conservation of Ancient Sites on the Silk Road* N. Agnew ed. (Los Angeles: Getty Conservation Institute, 1997), p. 146.

[7] Silver "Architectural Finishes of the Prehistoric Southwest," 1987, p. 153. Tests here cited were conducted in 1981.

[8] Paul M. Schwartzbaum, "The ICCROM Project for Conservation of Mural Paintings in the Rock Churches of the Goreme Valley (1971-1983),i *The Safeguard of the Rock-Hewn Churches of the Goreme Valley,"* (Rome: ICCROM, 1995), p. 187-202.

[9] R.S. Argumedo, "Mural Painting on Adobe Walls During Peruvian Colonial Times - Its Restoration and Conservation" In *Case Studies in the Conservation of Stone and Wall Paintings,* (London: IIC, 1986), p. 77.

[10] Morgan W. Phillips, "Experiences in the Use of Acrylic Plaster Adhesives," *Case Studies in the Conservation of Stone and Wall Paintings* N.S. Brommelle and P. Smith eds., (London: International Institute for conservation of Historic and Artistic Works, 1986), p. 34-38.

Adhesive	Use	Concentration	Location	User/Source	Comments
(ae) Acrysol ASE-60 (thickener) Fillers - Hydrated lime, flud coke, & microballoons	grouting			1986[10]	adhesive. The acrylic gel is much better suited for use in aqueous systems and is now recommended exclusively." p. 34
Rhoplex AC-33 (ae)	Earthen plaster reattachment and stabilization	not specified	Room 156, Aztec Ruin National Monument, NM	Silver, "The Conservation of Tempera Mural Paintings," 1990.[11]	Author states selection of ae emulsion over previous usage of pva was influenced by "some evidence that it will age better in the field." Otherwise, methods same as used in 1981 at MEVE.
Daraweld C (pva) 51% solids	Adobe preservation Mud plaster amendment	5% and 10%		Taylor, "An Evaluation," 1985-1990[12]	Erosion is in inverse proportion to resin concentration. Higher resin concentration - lower erosion. Slightly darker than unamended plasters. Discontinued usage due to product's relative impermeability.
Rhoplex E-330 (ae) 47% solids	Mud plaster amendment, repointing masonry walls	5% and 10%	Chaco Canyon, Aztec and Wuptaki Natl. Monuments	Taylor, "An Evaluation," 1985-1990[13]	Little erosion. Erosion in inverse proportion to resin concentration. Higher resin concentration - lower erosion.
Primal AC-33 (ae) Aerospray 70 Binder (pva)	Consolidation of loosely cemented agrillaceous conglomerate cliff rock and	1:10 in H_2O for laboratory testing 1:10, 1:20, 1:30 in H_2O on site testing	Mogao Grottoes, PRC	Li, Agnew and Lin, "Chemical Consolidation of Conglomerate," 1993[14]	Project concerns the exterior *in situ* use of the materials. The project tested, water absorption, freeze-thaw, water resistance (related to wvt) among others not directly related to this research.

[10] Silver, "The Conservation of Tempera Mural Paintings," p. 142.

[11] Michael R. Taylor, "An Evaluation of the New Mexico State Monuments Adobe Test Walls at Fort Selden," *Sixth International Conference on the Conservation of Earthen Architecture* (Los Angeles: Getty Conservation Institute, 1990), p. 385.

[12] Taylor, "An Evaluation of the New Mexico State Monuments," p. 385.

[13] Li, Agnew, and Lin, "Chemical Consolidation of Conglomerate and Sand at the Mogao Grottoes," *Conservation of Ancient Sites on the Silk Road* N. Agnew ed. (Los Angeles: Getty Conservation Institute, 1997), p. 194-212.

Adhesive	Use	Concentration	Location	User/Source	Comments
	loose sand				
BAF (Bonding Acrylic Fortifier) 54% solids [essentially Rhoplex E-330] (ae)	Adobe amendment	1:4 (BAF:H_2O) ppv	Montano Store, New Mexico State Monuments.	Selwitz & Caperton, "Chemical Stabilization," 1995[15]	
El Rey Superior 200 44% + 1% (ae)	Hydraulic lime grout formulations for lime plaster reattachment	10% in H_2O ppv w/ microballoons and lime		Matero and Bass, "Design and Evaluation," 1995[16]	Prewetting with 5% ae in H_2O
Rhoplex E-330 (ae)	Amended mud coatings for adobe walls	8% in H_2O 14% in H_2O	Fort Selden, New Mexico	Selwitz, "Saving the Fort Selden Ruins," 1995[17]	Other variables, sand:soil ratios, architectural function of the coating (capping or vertical veneer) and method of application (spray or trowel). Cracking of this coating was a major problem.
Latex acrylic polymer emulsion (ae)	Amended earthen mortars for the stabilization of megalithic monuments	30% latex (ae) 2% used ppw in mortar formulae	Genna Maria, Sardinia	Atzeni, Cabiddu, Massidda and Sanna, The Use of 'Stabilized Earth'," 1996[18]	
Rhoplex E-330 (ae)	Adobe amendment	6%, 12.5 %, 25% and 33%	Architectural Conservation Laboratory, UPenn	Hartzler, Acrylic-Modified Earthen Mortar, 1996[19]	Polymer % solids must be calculated in assessing performance. "A 33% ae in H_2O cannot overwhelm the natural characteristics of a soil." p. 54
El Rey Superior	Lime plaster	1:4 ae: H_2O w/	Fort Davis, TX	Hartzler, Historic	Injection grouting, two step prewetting a) H_2O

[15] Charles Selwitz and T. J. Caperton, "Chemical Stabilization of Adobe in the Restoration of the Montano Store," *APT Bulletin 26* 37-41.

[16] Frank G. Matero and Angelyn Bass, "Design and Evaluation of Hydraulic Lime Grouts for the Reattachment of Lime Plasters on Earthen Walls," *Conservation and Management of Archaeological Sites 1* (1995): 97-108.

[17] Charles Selwitz, "Saving the Fort Selden Ruins: the Use of a Composite Blend of Chemicals to Stabilize Fragile Historic Adobe," *Conservation and Management of Archaeological Sites 1* (1995): 109-116.

[18] C. Atzeni, M.G. Cabiddu, L. Massidda and U. Sanna, "The Use of 'Stabilized Earth' in the Conservation of Megalithic Monuments," *Conservation and Management of Archaeological Sites 1* (1996): 161-168.

[19] Robert Hartzler, *Acrylic-Modified Earthen Mortar*, (Santa Fe, New Mexico: United States National Park Service, 1996), p. 29

Adhesive	Use	Concentration	Location	User/Source	Comments
200 (ae)	reattachment with hydraulic lime grout	microballoons and lime		Building 15," 1997[20]	and b)10% ae

[20] Robert Hartzler, "Historic Building 15, Paint Consolidation, Plaster Stabilization and Overall Condition Assessment," August 18-29, 1997, unpublished report. Fort Davis National Historic Site. Fort Davis, Texas.

Comparison between polyvinyl acetate and acrylic emulsions[21]

	POLYVINYL ACETATE	ACRYLIC
pH and stability	acidic becoming more acidic over time	neutral to alkaline (basic) becoming more alkaline over time initially between 9-10 over time fell to 8-9.
Flexibility	less flexible	more flexible
Strength	more strength	less strength
UV sensitivity	less resistance to yellowing	greater resistance to yellowing[22]
Viscosity	more viscous	less viscous
Resistance to biological growth	good[23]	
Cross linking under UV	low tendency[24]	
Resolubility	high[25]	slightly lower[26]

[21] L. Down, M. A. MacDonald, J. Tétreault and R.S. Williams "Adhesive Testing at the Canadian Conservation Institute - An Evaluation of Selected Poly(Vinyl Acetate) and Acrylic Adhesives," *Studies in Conservation* 41 (1996): 19-44.

[22] C.V. Horie, *Materials for Conservation* (Oxford: Butterworth-Heinemann, 1995), p. 110.

[23] W. Boustead, "Conservation of Works of Art in Tropical and Sub-Tropical Zones," *Recent Advances in Conservation*, G. Thomson ed. (London: Butterworths, 1963), p. 75.

[24] R.L. Feller, "New Solvent-Type Varnishes," *Recent Advances in Conservation*, G. Thomson ed. (London: Butterworths, 1963), p. 174.

[25] E.H. Jones, "The Effect of Aging and Re-forming on the Ease of Solubility of Certain Resins," *Recent Advances in Conservation*, G. Thomson ed. (London: Butterworths, 1963), p. 81.

[26] Rutherford Gettens and George Stout, *Painting Materials; A Short Encyclopaedia* (New York: Dover Publications Inc., 1966), p. 3.

10.3 Product Specifications & Material Safety Data Sheets (MSDS)

February 9, 1998

**ROHM
IHAAS
COMPANY**

We at Rohm and Haas are undertaking a series of product rationalizations in an effort to streamline our operations. Our goal is to maintain a product line that offers:

- high quality products
- efficient customer services
- maximum production capacity and cost effectiveness

While we understand the frustrations involved when qualifying replacement products, we believe ultimately we will be a better supplier to you by offering higher value products and services.

We plan to discontinue selling **Rhoplex AC-33** by October 1, 1999. We suggest Rhoplex B-60A as a possible replacement. Rhoplex B-60A is similar in composition and solids, with the exception of a different biocide and without the incorporation of a rheology modifier. To achieve similar rheology with Rhoplex B-60A, we suggest incorporating 0.6% Acrysol G-111 or 0.5% Acrysol 6038A as a starting point.

Samples and literature are available for your evaluation. Please contact us at 800-423-0886 to order them. We regret any inconvenience this discontinuation may cause you.

Sincerely,

RHOPLEX® E-330
CEMENT MORTAR MODIFIER

RHOPLEX E-330 is a water dispersion of an acrylic polymer specifically designed for modifying Portland cement compositions. Important application areas include patching and resurfacing, floor underlayments, terrazzo flooring, spray and fill coats, precast architectural building panels, stucco, industrial cement floors, and highway and bridge deck repair. Additional information on cement modifiers is available in the technical notes for RHOPLEX MC-76, RHOPLEX MC-1834, and DRYCRYL DP-2903.

Performance Advantages

Durability and Strength

Cement mortars modified with RHOPLEX E-330 are hard, tough, and durable. Compared with unmodified mortars, polymer-modified mortars have superior flexural, adhesive, and impact strengths, as well as excellent abrasion resistance. They are especially useful where thin sections are desirable and where excessive vibration and heavy traffic are encountered.

Adhesion

RHOPLEX E-330-modified cement mortars have excellent adhesion to a variety of surfaces, such as concrete, masonry, brick, wood, and metals.

Resistance Properties

Cement mortars prepared with RHOPLEX E-330 are resistant to many industrial chemicals and have excellent resistance to ultraviolet light and heat. They dry to a uniform color with no tendency toward yellowing or discoloration.

Curing Advantages

For optimum physical properties, cement mortars modified with RHOPLEX E-330 should be air-cured at ambient temperature and relative humidity. Unlike unmodified mortars, which require laborious moist curing conditions for optimum strength properties, polymer-modified mortars should not be cured under these conditions.

Storage Advantages

RHOPLEX E-330 emulsion is
sediment-free and stable to a
minimum of five cycles of freezing at
-15°C and thawing at 25°C.

Typical Physical Properties
of Rhoplex E-330

Appearance	Milky, white liquid
Solids content, %	47 ± 0.5%
pH, when packed	9.3 to 10.2
Specific gravity	1 059
Pounds per gallon	8 8
Freeze/thaw stability	5 cycles
Minimum film-formation temperature	10°C to 12°C

RHOPLEX E-330 is stable for a
minimum of five cycles of freezing at
-15°C and thawing at 25°C.
However, in cold weather or after
prolonged storage, the emulsion
should be thoroughly stirred prior to
use to ensure a completely
homogeneous mixture.

Suggested starting point
formulations, test results and other
technical information for RHOPLEX
E-330 are noted in the Technical
Data Sheets for this product.

Safe Handling Information

Animal toxicity screening tests conducted on closely related analogs of RHOPLEX E-330 suggest that this product should be essentially non-toxic by single acute oral or dermal exposure and that it may also be a mild to moderate skin and eye irritant. In addition, many of the components of cement used in conjunction with RHOPLEX E-330 may also possess significant skin and eye irritation potential.

The Rohm and Haas Company maintains comprehensive and up-to-date Material Safety Data Sheets (MSDS) on all of its products. These sheets contain pertinent information

that you may need to protect your employees and customers against any known health or safety hazards associated with our products.

The Rohm and Haas Company recommends that you obtain copies of our Material Safety Data Sheets from your local Rohm and Haas representative on each of our products prior to its use in your facilities. We also suggest that you contact your supplier of other materials recommended for use with our products for appropriate health and safety precautions prior to their use.

ROHM & HAAS
PHILADELPHIA, PA 19106

ACRYLOID, ACRYSOL, ACUSOL, AQUAPLUS, DURAPLUS, ELASTENE, KATHON, MAINCOTE, PARAPLEX, RHOPLEX, ROBOND, ROPAQUE, ROVACE, SEALER-HEALER, SKANE, SKANE AND TAMOL are registered trademarks of Rohm and Haas Company or of its subsidiaries or affiliates.

These suggestions and data are based on information we believe to be reliable. They are offered in good faith, but without guarantee, as conditions and method of use of our products are beyond our control. We recommend that the prospective user determine the suitability of our materials and suggestions before adopting them on a commercial basis.

Suggestions for uses of our products or the inclusion of descriptive material from patents and the citation of specific patents in this publication should not be understood as recommending the use of our products in violation of any patent or as permission or license to use any patents of the Rohm and Haas Company.

SUGGESTED STARTING FORMULATIONS FOR RHOPLEX® E-330

Depending upon the particular application involved, a variety of cement mortar formulations shown below are useful as starting point systems.

The quantities of water cited in the formulations below should be considered as rough guides. Exact amounts depend on the type and brand of cement, particle size and moisture content of the sand, and on the other agents used in the mortar mix. Increasing amounts of RHOPLEX E-330 used in the cement mortar require decreasing amounts of water for a suitable workable consistency.

To prepare RHOPLEX E-330-modified mortar for evaluation, thoroughly premix the sand and cement. The RHOPLEX E-330, water and antifoamer should be blended together and added to the premixed sand and cement. The entire composition is mixed thoroughly for about two to four minutes. When preparing mortars for application in the field, a portion of the water should be withheld and added gradually to the modified mortar mixture until the desired consistency is obtained. This is necessary to avoid overly fluid compositions in those cases where

the sand is used in high moisture content or where other variables affect the amount of water to be used

Prior to testing, all mortar specimens were cured for 28 days. Unmodified mortars were prepared by both air-cured and wet-cured procedures, whereas the polymer-modified samples were all air-cured the Conditions employed for air and wet-curing are as follows:

AIR-CURING CONDITIONS
- 28 days at 25°C and 50% relative humidity

WET-CURING CONDITIONS
- 1 day at 25°C and 90% relative humidity

- 6 days water immersion at 25°C

- 7 days at 25°C and 50% relative humidity

- 7 days water immersion at 25°C

- 7 days at 25°C and 50% relative humidity

140

SUGGESTED STARTING FORMULATIONS

Ratio of Polymer Solids to Cement[a]	0.00	0.10	0.15	0.20
Material	Weight	Weight	Weight	Weight
Sand	300 0	300 0	300.0	300 0
Portland Cement (Type 1)	100 0	100 0	100 0	100 0
RHOPLEX® E-330 (47% Solids)	0 0	21 0	32 0	42 0
Defoamer[b]	0 0	0 10[c]	0 15[c]	0 20[c]
Water	48 0	29 0	20 0	11 0
Ratio of Water to Cement[a]	0 48	0 40	0 37	0 35
Ratio of Sand to Cement[a] 3 0	3 0	3 0		3 0

(a)   By weight

(b)   Recommended Defoamers

Nopco NXZ (100% active) - Henkel Corporation, Ambler, PA 19002-3491

GE Antifoam #60 (30% active) - General Electric Co., Silicone Products Dept., Waterford, NY

(c)   Suggested minimum of 1% based on polymer solids using 100% active defoamer More may be added, if necessary, to maximize wet density

FORMULATION GUIDE WITH RHOPLEX E-330

ACRYLIC SPACKLE COMPOUND FORMULATION (JK-789C)

	Pounds
Rhoplex E-330 (47%)	228
Tamol 850 (30%)	2
Ethylene glycol	48
Acrysol ASE-60 (28%)	4
#8 Marble dust (Georgia Marble Co., Tate, Ga.)	980
Titanium dioxide (Ti-Pure R-901)	12
	1274

Mixing Procedure

Using a double-bladed sigma-type mixer:

1) Premix the liquid ingredients and charge about 75% to the mixer.

2) Add sufficient #8 Marble dust and TiO_2 to make a thick paste (approximately 70% of the dry ingredients) while mixing.

3) Add the remainder of the liquids and mix thoroughly.

4) Add the remainder of the dry ingredients.

5) Cover mixer tightly and mix for about one hour.

6) About five minutes before the completion of the batch, add 0.3 to 0.4 lbs. of Nopco NXZ defoamer (0.25% based on polymer solids).

ACRYSOL, RHOPLEX and TAMOL are trademarks of Rohm and Haas Company or of its subsidiaries or affiliates, and are intended to designate goods marketed in North and South America, the same goods may be marketed in other countries, generally under other Company trademark designations.

TESTING OF MORTAR SPECIMENS WITH RHOPLEX® E-330

TEST		METHOD	ASTM STANDARD
1	Tensile Strength	1" thick briquettes	C-190-85
2	Compressive Strength	2" cubes	C-109-88
3	Flexural Strength	Flexural bar	C-348-86
4	Set Time[1]	Modified Vicat needle	C-191-82
5	Shear Bond Strength[2]	Rohm and Haas method	
6	Impact Strength[3]	Gardner Impact Tester IG-1115	
7	Abrasion Resistance[4]	Taber Abrader	
8	Emulsion Polymer Minimum Film Formation Temperature	MFT Bar	D-2354-86

1   Set Time - The ASTM C-191-82 test procedure was modified in that a conical rather than a vicat needle was employed. Needle penetration was determined on the unexposed rather than the exposed surface of the truncated cone of mortar to measure through set rather than surface hardening.

2   Shear Bond Strength - A 2" x 2" x 0.5" polymer-modified cement mortar patch is centrally cast and cured on a 5" x 2.5" x 0.75" cured unmodified concrete base piece. A suitable plunger applies a load at 0.05 inches per minute to the patch until failure occurs. The shear bond strength (psi) equals the load in pounds divided by the interfacial area of the patch in square inches.

3   Impact Strength - A Gardner Impact Tester IG-1115 mounted on a 1.25 inch thick slab of Chemstone is employed. Testing is done on a 0.75 inch thick wood table top supported on iron legs. Mortar specimens are prepared 5.0" x 2.375" x 0.75". Specimens are impacted until break in one of the following series of increments: 2, 4, 6, 8, 10...inch/lbs; 2, 6, 10, 14, 18...inch/lbs; 6, 12, 18, 24...inch/lbs. An average of five samples is taken.

4   Abrasion Resistance - A model 174 Taber Abrader equipped with an H-22 calibrade abrasion wheel is employed. Cylindrical mortar specimens (4 5/8" x 1/2") are prepared and are subject to 1,000 cycles under a load of 1,000 grams and abrasion resistance is reported as the percent weight loss of the samples.

Polymer Modification

In general, cement mortars modified with RHOPLEX E-330 and air-cured have superior flexural, shear bond adhesion, and impact strengths when compared to moist cured, unmodified cement mortars. In addition, polymer modification results in improved abrasion resistance and comparable tensile and compressive strengths when compared to unmodified mortars. This information is presented in the table below and the figures represent average values of a large number of samples tested.

PHYSICAL STRENGTH PROPERTIES OF PORTLAND CEMENT MORTAR

Ratio of RHOPLEX E-330 Polymer Solids to cement weight	0.0	0.10	0.15	0.20
Ratio of water to cement[1]	0.48	0.40	0.37	0.35
Tensile Strength, psi				
28 day air-cure	235	530	615	855
28 day wet-cure	535	-	-	-
28 day air-cure + 7 day water soak	310	330	350	490
Compressive Strength, psi				
28 day air-cure	2390	5450	5715	5690
28 day wet-cure	5795	-	-	-
28 day air-cure + 7 day water soak	4420	4700	5125	5460
Flexural Strength, psi				
28 day air-cure	610	1355	1585	1835
28 day wet-cure	1070	-	-	-
28 day air-cure + 7 day water soak	735	950	1020	1050
Shear Bond Adhesion, psi [2]				
28 day air-cure	45(A)	>500(C)	>650(C)	>550(C)
28 day wet-cure	185(A)	-	-	-
28 day air-cure + 7 day water soak	140(A)	290(C)	300(C)	330(C)
Impact Strength, inch/lbs				
28 day air-cure	6	12	16	22
28 day wet-cure	7	-	-	-
28 day air-cure + 7 day water soak	9	11	13	18
Abrasion Resistance, percent weight loss [3]				
28 day air-cure	23 8	1 70	1.15	1 57
28 day wet-cure	5 07	-	-	-

(1) The water content of the mortars was adjusted to provide equivalent workability, ie ,the polymer-modified mortars were prepared at water contents necessary to give the same slump diameter which was equivalent in workability to the unmodified mortar controls at 48 percent water content. This procedure is described in the ASTM "Flow Rate" test #C-230-83

(2) Adhesive failure indicated by (A) Cohesive failure indicated by (C)

(3) Lower values indicate better abrasion resistance

83D1b
Page 9

ROHM AND HAAS COMPANY

August 1995
Printed in U S A

145

Density

The graph below shows the effect of density variation on the tensile strength of cement mortar modified with RHOPLEX E-330. As the density increases, so does the tensile strength. Similarly, one can improve the compressive, flexural, impact and adhesive strengths by increasing the mortar density. Therefore, when modifying cement mortars with RHOPLEX E-330 it is important to minimize the air entrainment due to foaming. By using an appropriate amount of antifoamer it is possible to get high density, polymer-modified mortars with excellent strength properties. In general, the wet density of a latex-modified cement mortar should be at least the same as unmodified mortar. In most cases the wet density will be 2.0 g/cm³ or higher.

TENSILE STRENGTH vs DENSITY

RHOPLEX E-330/Cement = 0.15
Water/Cement = 0.35
Sand/Cement = 3/1
7 Day Air-Cure

146
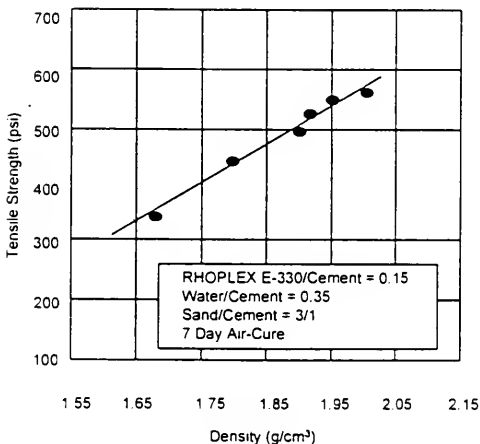
VARIABLES AFFECTING CEMENT MORTAR PROPERTIES USING RHOPLEX® E-330

Length of Cure

All cement mortars continue to cure with the passage of time. It is believed that it takes about 28 days for both a polymer-modified and an unmodified cement mortar to obtain approximately 90 percent of their ultimate physical properties. The graph below shows the increase in tensile strengths of RHOPLEX E-330-modified and unmodified mortars as a function of time. Similar behavior also occurs in other physical strength properties.

Curing Conditions

To obtain maximum physical strength properties, RHOPLEX E-330-modified cement mortars should be air-cured at ambient temperature and relative humidity, avoiding the use of moist curing techniques, a laborious procedure used with unmodified mortars to obtain optimum properties.

TENSILE STRENGTH vs DAYS AIR-CURE

Sand/Cement = 3/1

RHOPLEX E-330/CEMENT = 0.20
Water/Cement = 0.30

Polymer/Cement = 0
Water/Cement = 0.45

147

Water Level

The amount of water that should be used in a cement mortar depends on the type and brand of cement used, the particle size and moisture content of the sand used, the additional ingredients of the mortar mix, and the working consistency desired for the particular application. As a general rule, water should be held to the lowest amount needed to achieve a suitable working consistency. By using a minimum amount of water maximum strength properties are obtained.

The effect of the water/cement ratio on tensile strength at a constant polymer level is shown in the graph below.
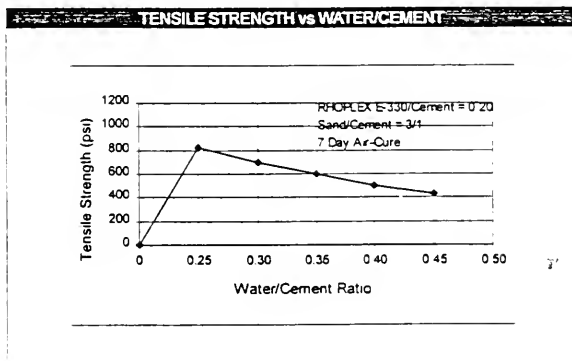
TENSILE STRENGTH vs WATER/CEMENT

RHOPLEX E-330/Cement = 0.20
Sand/Cement = 3/1
7 Day Air-Cure

Tensile Strength (psi) vs Water/Cement Ratio

83D1b   ROHM AND HAAS COMPANY   August 1995
Page 12   Printed in U.S.A.

148

PRODUCT SPECIFICATIONS

RHOPLEX E-330 (6-6580)

Appearance, as-is
    visual
                                  Milk-white fluid, free
of coagulated gum and
visible impurity.

Solids content, % by wt.   46.5 - 47.5
    Dry 0.6 gram at 150°C
    for 20 minutes in a
    forced draft oven.

pH   ASTM E-70   9.30 - 10.20

Viscosity, as is, cps   5 - 55
    Brookfield LV
    spindle #1, 60 rpm, 25°C

Gel particles on 100 mesh   0.05, maximum
    grams dry gel/quart

December 17, 1997

MATERIAL SAFETY DATA SHEET RHOPLEX E-330

Rohm and Haas Company
100 Independence Mall West
Philadelphia, PA 19106-2399

HEALTH EMERGENCY   215-592-3000
SPILL EMERGENCY   215-592-3000
CHEMTREC   800-424-9300

PRODUCT IDENTIFICATION

RHOPLEX E-330 Emulsion

MSDS date   07/11/90

Rohm and Haas
    Hazard Rating   Scale
                         4=EXTREME
Toxicity   1   3=HIGH
Fire   0   2= MODERATE
Reactivity   0   1=SLIGHT
Special   0=INSIGNIFICANT

COMPONENT INFORMATION

No.		CAS REG NO.	AMT.(%)
1	Acrylic polymer	Not Hazardous	46-48
2	Individual residual monomers	Not Required	less than 0.1
3	Ammonia	7664-41-7	0.2 MAX.
4	Water	7732-18-5	52-54

EMERGENCY RESPONSE INFORMATION

FIRST AID PROCEDURES

Inhalation
Move subject to fresh air.

Eye Contact
Flush eyes with a large amount of water for at least 15 minutes.
Consult a physician if irritation persists.

Skin Contact
Wash affected skin areas thoroughly with soap and water. Consult
a physician if irritation persists.

Ingestion
If swallowed, give 2 glasses of water to drink. Consult a
physician. Never give anything by mouth to an unconscious person.

FIRE FIGHTING INFORMATION

Unusual Hazards

The information herein is given in good faith, but no warranty, express or

MATERIAL SAFETY DATA SHEET RHOPLEX E-330

Material can splatter above 100C (212F). Polymer film can burn.

Extinguishing Agents
Use extinguishing media appropriate for surrounding fire.

Personal Protective Equipment
Wear self-contained breathing apparatus (pressure-demand MSHA/NIOSH
approved or equivalent) and full protective gear.

SPILL OR LEAK HANDLING INFORMATION

Personal Protection
Appropriate protective equipment must be worn when handling a spill
of this material. See the PERSONAL PROTECTION MEASURES Section for
recommendations. If exposed to material during clean-up operations,
see the FIRST AID PROCEDURES Section for actions to follow.

Procedures
Keep spectators away. Floor may be slippery; use care to avoid
falling. Contain spills immediately with inert materials (e.g.
sand, earth). Transfer liquids and solid diking material to
separate suitable containers for recovery or disposal.
CAUTION: Keep spills and cleaning runoff out of municipal sewers
and open bodies of water.

HAZARD INFORMATION

HEALTH EFFECTS FROM OVEREXPOSURE

Primary Routes of Exposure
              Inhalation
              Skin Contact

Inhalation
Inhalation of vapor or mist can cause the following:
- headache, nausea . irritation of nose, throat, and lungs

Eye Contact
Direct contact with material can cause the following:
- slight irritation

Skin Contact
Prolonged or repeated skin contact can cause the following:
- slight skin irritation

FIRE AND EXPLOSIVE PROPERTIES

Flash Point   Noncombustible
Auto-ignition temperature   Not Applicable
Lower explosive limit   Not Applicable
Upper explosive limit   Not Applicable

REACTIVITY INFORMATION

The information herein is given in good faith, but no warranty, express or
implied, is made.

MATERIAL SAFETY DATA SHEET RHOPLEX E-330

Instability
This material is considered stable. However, avoid temperatures
above 177C/350F, the onset of polymer decomposition. Thermal
decomposition is dependent on time and temperature.

Hazardous Decomposition Products
Thermal decomposition may yield acrylic monomers.

Hazardous Polymerization
Product will not undergo polymerization.

Incompatibility
There are no known materials which are incompatible with this product.

ACCIDENT PREVENTION INFORMATION

COMPONENT EXPOSURE INFORMATION

Component Information

No.		CAS-REG-NO.	AMT (%)
1	Acrylic polymer	Not Hazardous	46-48
2	Individual residual monomers	Not Required	less than 0.1
3	Ammonia	7664-41-7	0.2 MAX
4	Water	7732-18-5	52-54

Exposure Limit Information

Component		ROHM AND HAAS		OSHA		ACGIH	
No.	Units	TWA	STEL	TWA	STEL	TLV	STEL
1		None	None	None	None	None	None
2		a	a	a	a	a	a
3	PPM	25	35	None	35	25	35
4		None	None	None	None	None	None

a   Not Required

PERSONAL PROTECTION MEASURES

Respiratory Protection
None required if airborne concentrations are maintained below the
TWA/TLV's listed in the COMPONENT EXPOSURE INFORMATION Section.
For airborne concentrations up to 10 times the TWA/TLV's listed in
the COMPONENT EXPOSURE INFORMATION Section wear a MSHA/NIOSH
approved (or equivalent) half-mask, air-purifying respirator.
Air-purifying respirators should be equipped with organic
vapor cartridges.

Eye Protection
Use chemical splash goggles (ANSI Z87.1 or approved equivalent).

Hand Protection
The glove(s) listed below may provide protection against

The information herein is given in good faith, but no warranty, express or
implied, is made.

MATERIAL SAFETY DATA SHEET RHOPLEX E-330

permeation. Gloves of other chemically resistant materials may not
provide adequate protection:
- Neoprene

FACILITY CONTROL MEASURES

Ventilation
Use local exhaust ventilation with a minimum capture velocity of
100 ft/min. (30 m/min.) at the point of vapor evolution. Refer to
the current edition of Industrial Ventilation: A Manual of
Recommended Practice published by the American Conference of
Governmental Industrial Hygienists for information on
the design, installation, use, and maintenance of exhaust systems.

Other Protective Equipment
Facilities storing or utilizing this material should be equipped
with an eyewash facility.

STORAGE AND HANDLING INFORMATION

Storage Conditions
Keep from freezing; material may coagulate. The minimum recommended
storage temperature for this material is 1C/34F. The maximum
recommended storage temperature for this material is 49C/120F.

Handling Procedures
Monomer vapors can be evolved when material is heated during
processing operations   See FACILITY CONTROL MEASURES Section for
types of ventilation required.

SUPPLEMENTAL INFORMATION

TYPICAL PHYSICAL PROPERTIES

Appearance	Milky
Color	White
State	Liquid
Odor Characteristic	Ammonia odor
pH	9.5-10.5
Viscosity	50 CPS Maximum
Specific Gravity (Water = 1)	1.0-1.2
Vapor Density (Air = 1)	less than 1 Water
Vapor Pressure	17 mm Hg at 20 Deg C/ 68 Deg F Water
Melting point	0 Deg C / 32 Deg F Water
Boiling point	100 Deg C / 212 Deg F
Solubility in water	Dilutable
Percent Volatility	52-54% Water
Evaporation rate (BAc = 1)	less than 1 Water

TOXICITY INFORMATION

Acute Data

The information herein is given in good faith, but no warranty, express or
implied, is made.

MATERIAL SAFETY DATA SHEET RHOPLEX E-330

The information shown in the HEALTH EFFECTS FROM OVEREXPOSURE
Section is based on the toxicity profiles for a number of acrylic
emulsions that are compositionally similar to this product.
Typical data are:

Dermal LD50 - rabbit: greater than 5000 mg/kg
Oral LD50 - rat: greater than 5000 mg/kg
Skin irritation - rabbit: practically non-irritating
Eye irritation - rabbit: inconsequential irritation

WASTE DISPOSAL

Procedure
Coagulate the emulsion by the stepwise addition of ferric chloride
and lime. Remove the clear supernatant and flush to a chemical
sewer. Incinerate liquid and contaminated solids in accordance with
local, state, and federal regulations.

REGULATORY INFORMATION

WORKPLACE CLASSIFICATIONS
This product is considered non-hazardous under the OSHA Hazard
Communication Standard (29CFR 1910.1200).
This product is not a 'controlled product' under the Canadian
Workplace Hazardous Materials Information System (WHMIS).

TRANSPORTATION CLASSIFICATIONS
US DOT Hazard Class   NONREGULATED

EMERGENCY PLANNING & COMMUNITY RIGHT-TO-KNOW (SARA TITLE 3)

Section 311/312 Categorizations (40CFR 370)
This product is not a hazardous chemical under 29CFR-1910.1200, and
therefore is not covered by Title III of SARA.

Section 313 Information (40CFR 372)
This product does not contain a chemical which is listed in Section
313 above de minimis concentrations.

CERCLA INFORMATION (40CFR 302.4)
Releases of this material to air, land, or water are not reportable
to the National Response Center under the Comprehensive
Environmental Response, Compensation, and Liability Act; (CERCLA)
or to state and local emergency planning committees under the
Superfund Amendments and Reauthorization Act
(SARA) Title III Section 304.

RCRA INFORMATION
When this product becomes a waste, it is classified as a
non-hazardous waste under criteria of the Resource Conservation and
Recovery Act (40 CFR 261).

CHEMICAL CONTROL LAW STATUS
All components of this product are listed or are excluded from
listing on the U.S. Toxic Substances Control Act (TSCA) Chemical

The information herein is given in good faith, but no warranty, express or
implied, is made.

MATERIAL SAFETY DATA SHEET RHOPLEX E-330

Substance Inventory.

STATE RIGHT-TO-KNOW LAWS
Any material listed as "Not Hazardous" in the CAS REG NO column of
the COMPONENT INFORMATION Section of this MSDS is trade secret
under the provisions of the Pennsylvania Worker and Community
Right-to-Know Act.

"RHOPLEX" is a trademark of Rohm and Haas Company or one of its
subsidiaries or affiliates.

ABBREVIATIONS:
ACGIH =American Conference of Governmental Industrial Hygienists
OSHA   = Occupational Safety and Health Administration
TLV   = Threshold Limit Value
PEL   = Permissible Exposure Limit
TWA   = Time Weighted Average
STEL   = Short-Term Exposure Limit
BAc   = Butyl acetate

The information herein is given in good faith, but no warranty, express or
implied, is made.

Sodium Polyacrylate
Thickener

Acrysol® 6038A

GENERAL INFORMATION

Acrysol 6038A is a sodium polyacrylate thickener which demonstrates predictable viscosity response and imparts excellent efficiency in latex compounds. The thickener is easily dispersed at full strength and is activated in an alkaline system. Latices thickened with Acrysol 6038A demonstrate a short, smooth flow This product's good dispersing properties improve the filler acceptance of highly loaded compounds.

Formulations using Acrysol 6038A are especially stable to viscosity drift with time in a wide variety of compounds.

TYPICAL PROPERTIES

The following properties are typical and should not be considered specifications.

Polymer Type	Sodium Polyacrylate
Appearance	Clear, light-amber soln.
Solids, %	13.5
pH	9.0
Brookfield RVF	
Viscosity, cps	21,000
(#2 spindle.	
20 RPM @ 25°C)	
Density lbs/US gal	8.9
gm/ml	1.1
Freeze/Thaw	
Stability	Protect from freezing

COMPLIANCE

Acrysol 6038A complies with applicable requirements of FDA Food Additive Regulation 21CFR 175 105, 176 170*, 175 300** and 176 180 We recommend that you make your own determination on the suitability of Acrysol 6038A for your particular application We also recommend that you periodically verify the regulatory status of Acrysol 6038A.

* *Provided the food contact surface of the paper and paperboard complies with the extractives limitations prescribed in Paragraph (c) of 21CFR 176 170*

** *Provided that Acrysol 6038A is cleared as the basic polymer and as a modifier for oils and alkyds, including polyesters*

88A129

SAFE HANDLING INFORMATION

This product contains materials which have potential for skin and eye irritation. Therefore, this product must be handled with care and with strict adherence to industrial hygiene practices. Consult the Material Safety Data Sheet before using this product.

MATERIAL SAFETY DATA SHEETS

Rohm and Haas Material Safety Data Sheets (MSDS) contain pertinent information that you may need to protect your employees and customers against any known health or safety hazards associated with our products.

Under the OSHA Hazard Communication Standard, workers must have access to and understand MSDS on all hazardous substances to which they are exposed. Thus, it is important that you provide appropriate training and information to your employees and make sure they have available to them MSDS on any hazardous products in their workplace.

Rohm and Haas Company sends MSDS on non-OSHA-hazardous as well as OSHA-

hazardous products to both the "bill to" and "ship to" locations of all our customers upon initial shipment (including samples) of all our products (whether or not they are considered OSHA-hazardous). If you do not have access to one of these MSDS, please contact your local Rohm and Haas representative for an additional copy. Updated MSDS are sent upon revision to all customers of record.

MSDS should be obtained from your suppliers of other materials recommended in this bulletin.

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PRODUCT SPECIFICATIONS

ACRYSOL 6039A (7-3339)

Appearance, as-is Visual	White fluid, free of coagulated gum and visible impurity.
Solids content, % by wt. Dry 0.6 gram at 150°C for 20 minutes in a forced draft oven.	13.0 – 13.5
pH ASTM E-70	9.00 – 9.50
Viscosity, as is, cps Brookfield RV spindle #6, 20 rpm, 25°C	18000 – 25000

March 9, 1994

MATERIAL SAFETY DATA SHEET ACRYSOL 603BA EMULSION

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1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

ACRYSOL 603BA Emulsion

COMPANY IDENTIFICATION
Rohm and Haas Company
100 Independence Mall West
Philadelphia, PA 19106-2399

MSDS Date: 08/29/94

EMERGENCY TELEPHONE NUMBERS
HEALTH EMERGENCY : 215-592-3000
SPILL EMERGENCY   : 215-592-3000
CHEMTREC   : 800-424-9300

ACRYSOL is a trademark of Rohm and Haas Company or one of its
subsidiaries or affiliates.

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2. COMPOSITION/INFORMATION ON INGREDIENTS

No.		CAS REG NO.	WEIGHT (%)
1	Acrylic polymer, sodium salt	Not Hazardous	10-11
2	Individual residual monomers	Not Required	less than 0.1
3	Methanol	67-56-1	1-5
4	Water	7732-18-5	86-87

See SECTION 8, Exposure Controls / Personal Protection

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3. HAZARDS IDENTIFICATION

Primary Routes of Exposure
            Inhalation
            Eye Contact
            Skin Contact

- Inhalation
Inhalation of vapor or mist can cause the following:
- headache - nausea - irritation of nose, throat, and lungs.

Eye Contact
Direct contact with material can cause the following:
- slight irritation

Skin Contact
Prolonged or repeated skin contact can cause the following:
- slight skin irritation

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4. FIRST AID MEASURES

Inhalation
Move subject to fresh air.

Eye Contact

The information herein is given in good faith, but no warranty, express or
implied, is made.

MATERIAL SAFETY DATA SHEET ACRYSOL 6038A EMULSION

Flush eyes with a large amount of water for at least 15 minutes.
Consult a physician if irritation persists.

Skin Contact
Wash affected skin areas thoroughly with soap and water. Consult
a physician if irritation persists.

Ingestion
If swallowed, give 2 glasses of water to drink. Consult a
physician. Never give anything by mouth to an unconscious person.

5. FIRE FIGHTING MEASURES

Flash Point   Noncombustible
Auto-ignition Temperature   Not Applicable
Lower Explosive Limit   Not Applicable
Upper Explosive Limit   Not Applicable

Unusual Hazards
Material can splatter above 100C/212F. Dried product can burn.

Extinguishing Media
Use extinguishing media appropriate for surrounding fire.

Personal Protective Equipment
Wear self-contained breathing apparatus (pressure-demand MSHA/NIOSH
approved or equivalent) and full protective gear.

6. ACCIDENTAL RELEASE MEASURES

Personal Protection
Appropriate protective equipment must be worn when handling a spill
of this material. See SECTION 8, Exposure Controls/Personal
Protection, for recommendations. If exposed to material during
clean-up operations, see SECTION 4, First Aid Measures, for actions
to follow.

Procedures
Keep spectators away. Floor may be slippery; use care to avoid
falling. Contain spills immediately with inert materials (e.g.
sand, earth). Transfer liquids and solid diking material to
separate suitable containers for recovery or disposal.
  CAUTION: Keep spills and cleaning runoff out of municipal sewers
and open bodies of water.

7. HANDLING AND STORAGE

Storage Conditions
Keep from freezing; material may coagulate. The minimum
recommended storage temperature for this material is 1C/34F. The
maximum recommended storage temperature for this material is
49C/120F.

The information herein is given in good faith, but no warranty, express or
implied, is made.

MATERIAL SAFETY DATA SHEET ACRYSOL 6038A EMULSION

Handling Procedures
Monomer vapors can be evolved when material is heated during
processing operations. See SECTION 8, Exposure Controls/Personal
Protection, for types of ventilation required.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Exposure Limit Information
No.		CAS REG NO	WEIGHT (%)
	Acrylic polymer, sodium salt	Not hazardous	10-11
	Individual residual monomers	Not Required	less than 0.1
	Methanol	67-56-1	1-5
	Water	7732-18-5	86-87

Component	ROHM AND HAAS		OSHA		ACGIH	
No.	Units	TWA	STEL	TWA	STEL	TWA	STEL
1		None	None	None	None	None	None
2		4	4	3	3	3	3
3	ppm	200Skin	250Skin	200Skin	250 Skin	200 Skin	250Skin
4		None	None	None	None	None	None

3   Not Required

Respiratory Protection
A respiratory protection program meeting OSHA 1910.134 and ANSI
Z88.2 requirements must be followed whenever workplace conditions
warrant a respirator's use. None required if airborne
concentrations are maintained below the exposure limit listed in
'Exposure Limit Information'.

Up to 1000 ppm organic vapor: Wear a MSHA/NIOSH approved (or
equivalent) full-facepiece, air-purifying respirator,
                            OR

full facepiece, air line respirator in the demand mode.

Above 1000 ppm organic vapor or Unknown: wear a MSHA/NIOSH approved
(or equivalent) self-contained breathing apparatus in the
pressure demand mode,
                    OR
MSHA/NIOSH approved (or equivalent) full-facepiece, airline
respirator in the pressure demand mode with emergency
escape provision.
Air-purifying respirators should be equipped with organic vapor
cartridges and dust and mist filters.

Eye Protection
Use chemical splash goggles (ANSI Z87.1 or approved equivalent).

Hand Protection
The glove(s) listed below may provide protection against
permeation. Gloves of other chemically resistant materials may not
provide adequate protection:
 - Neoprene

Engineering Controls (Ventilation)

The information herein is given in good faith, but no warranty, express or
implied, is made.

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MATERIAL SAFETY DATA SHEET ACRYSOL 6038A EMULSION

Use local exhaust ventilation with a minimum capture velocity of
100 ft/min. (0.5 m/sec.) at the point of vapor evolution. Refer
to the current edition of Industrial Ventilation: A Manual of
Recommended Practice published by the American Conference of
Governmental Industrial Hygienists for information on the design,
installation, use, and maintenance of exhaust systems.

Other Protective Equipment
Facilities storing or utilizing this material should be equipped
with an eyewash facility.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance	Milky
Color	White
State	Liquid
Odor Characteristic	Acrylic odor
pH	8.0 to 9.5
Viscosity	15000 to 25000 CPS
Specific Gravity (Water = 1)	1.0 to 1.2
Vapor Density	less than 1 Water
Vapor Pressure	17 mm Hg @ 20 Deg C/68 DegF Water
Melting Point	0 DegC/32 DegF Water
Boiling Point	100 DegC/212 DegF Water
Solubility in Water	Dilutable
Percent Volatility	86 to 87 % Water and alcohol
Evaporation Rate	less than 1 Water

See SECTION 5, Fire Fighting Measures

10. STABILITY AND REACTIVITY

Instability
This material is considered stable. However, avoid temperatures
above 177C/350F, the onset of polymer decomposition. Thermal
decomposition is dependent on time and temperature.

Hazardous Decomposition Products
Thermal decomposition may yield acrylic monomers.

Hazardous Polymerization
Product will not undergo polymerization.

Incompatibility
There are no known materials which are incompatible with this product.

11. TOXICOLOGICAL INFORMATION

Acute Data
No toxicity data are available for this material. The toxicity
data listed are typical.
Oral LD50 - rat: greater than 5000 mg/kg
Dermal LD50 - rabbit: greater than 5000 mg/kg

The information herein is given in good faith, but no warranty, express or
implied, is made.

MATERIAL SAFETY DATA SHEET ACRYSOL 603RA EMULSION

Skin irritation - rabbit: practically non-irritating
Eye irritation - rabbit: inconsequential irritation

12. ECOLOGICAL INFORMATION

No Applicable Data

13. DISPOSAL CONSIDERATIONS

Procedure
Coagulate the emulsion by the stepwise addition of ferric chloride
and lime. Remove the clear supernatant and flush to a chemical
sewer. Incinerate liquid and contaminated solids in accordance with
local, state, and federal regulations

14. TRANSPORT INFORMATION

US DOT Hazard Class   . . . . NONREGULATED

15. REGULATORY INFORMATION

Workplace Classification
This product is considered non-hazardous under the OSHA Hazard
Communication Standard (29CFR 1910.1200).
This product is not a 'controlled product' under the Canadian
Workplace Hazardous Materials Information System (WHMIS).

SARA TITLE 3: Section 311/312 Categorizations (40CFR 370)
This product is not a hazardous chemical under 29CFR 1910.1200
and therefore is not covered by Title III of SARA.

SARA TITLE 3: Section 313 Information (40CFR 372)
This product contains a chemical which is listed in Section 313 at
or above de minimis concentrations. The following listed chemicals
are present: (Quantity present is found elsewhere on this MSDS.)
  - Methanol (67-56-1)

CERCLA Information (40CFR 302.4)
This material is regulated under the Comprehensive Environmental
Response, Compensation and Liability Act (CERCLA) and the Superfund
Amendments and Reauthorization Act (SARA) Title III Section 304.
This material is or contains chemical(s) listed in 40 CFR Table
302.4 or nondesignated RCRA ICR substance(s).
(Nondesignated ICR substances apply to materials that will not be
reused.) The Reportable Quantity(s) (RQ) are listed below.
Releases in excess of its reportable quantity must be reported to
the National Response Center (1-800-424-8802) and to the
appropriate state and local emergency response organizations.
  Methanol (67-56-1) 5000lbs.

Waste Classification

The information herein is given in good faith, but no warranty, express or
implied, is made.

163

MATERIAL SAFETY DATA SHEET ACRYSOL SG39A EMULSION

When a decision is made to discard this material, it
does not meet RCRA's characteristic definition of ignitability,
corrosivity, or reactivity, and is not listed in 40 CFR 261.33.
The toxicity characteristic (TC), however, has not been evaluated
by the Toxicity Characteristic Leaching Procedure (TCLP).

United States
All components of this product are in compliance with the inventory
listing requirements of the U.S. Toxic Substances Control Act
(TSCA) Chemical Substance Inventory.

Pennsylvania
Any material listed as "Not Hazardous" in the CAS REG No. column
of SECTION 2, Composition/Information On Ingredients, of this MSDS
is a trade secret under the provisions of the Pennsylvania Worker
and Community Right to Know Act.

16. OTHER INFORMATION

Rohm and Haas   Scale
Hazard Rating
  Toxicity   1   4-EXTREME
  Fire   0   3-HIGH
  Reactivity 0   2-MODERATE
  Special   -   1-SLIGHT
                                 0-INSIGNIFICANT
  Ratings are based on Rohm and Haas guidelines, and are intended
  for internal use.

The information herein is given in good faith, but no warranty, express or
implied, is made.

PRODUCT SPECIFICATIONS

RHOPLEX AC-33 (6-5197)

Appearance, as-is visual	Milk-white fluid, free of coagulated gum and visible impurity.
Solids content, % by wt. Dry 0.5 gram at 150°C for 20 minutes in a forced draft oven.	46.0 - 47.0
pH ASTM E-70	9.00 - 9.90
Viscosity, as-is, cps Brookfield LV spindle #3, 12 rpm, 25°C	1500 - 7500
Gel particles on 100 mesh grams dry gel/quart	0.05, maximum
Gel particles on 325 mesh grams dry gel/quart after passing through 100 mesh	0.10, maximum

November 22, 1994

3M
Scotchlite™
Glass Bubbles
K Series

Features, Advantages, and Benefits

Feature	Advantage	Benefit
Published Product Specification	Measurable and consistent product parameters	Predictable performance
Low Density	Reduced composite weight	Potential freight savings
Spherical Shape (minimum surface area to volume ratio)	Low resin demand	Lower viscosity at equal volume loading
		Potential for more filler at equal viscosity
		Reduced resin usage may result in reduced shrinkage
		Sprayable, castable and moldable
		Blends readily into compounds
Chemically Stable Glass	Low alkalinity	Compatible with most resins
		Stable shelf life and viscosity
	Non-combustible	Reduced Fire Hazard
	Non-porous	Does not absorb resin
	Excellent water resistance	Can be formulated to make stable emulsions
Variety of Product Types	Flexibility to meet varied product/processing requirements	Can select Glass Bubbles to meet many required parameters
Specially Formulated Hollow Glass Spheres	High strength to weight ratio	Able to survive processing
	Stable voids	Low thermal conductivity
		Low dielectric constant

Scotchlite™ Glass Bubbles
K Series

Typical Properties	Note: The following product information and data should be considered representative or typical only and should not be used for specification purposes.

Isostatic Crush Strength: See Product Specifications, paragraph B.

Density: See Product Specifications, paragraph C.

Chemical Resistance: In general, the chemical properties of Scotchlite Glass Bubbles resemble those of a soda-lime borosilicate glass.

Packing Factor: Varies from 55% to 68%
(Ratio of bulk density to true particle density.)

Oil Absorption: 31-36 g oil/100 cc of Glass Bubbles, per ASTM D1483.

Thermal Properties:

    A. Conductivity: 0.4 to 1.4 (Btu x in.) / (hr. x ft.^{2} x °F) at 32°F (0°C) based on theoretical calculations. Conductivity increases with temperature and product density. The thermal conductivity of a composite will depend on the matrix material and volume loading of glass bubbles.

    B. Stability: Appreciable changes in bubble properties may occur above 600°C depending on temperature and duration of exposure.

Flotation: See Product Specifications, paragraph F.

Volatile Content: See Product Specifications, paragraph G.

Alkalinity: See Product Specifications, paragraph E.

pH: Since Glass Bubbles are a dry powder, pH is not defined. The pH effect will be determined by the alkalinity as specified in paragraph E of the Specification. When glass bubbles are mixed with de-ionized water at 5 volume percent loading, the resulting pH of the slurry is typically 9.1 to 9.9, as measured by a pH meter.

Dielectric Constant: 1.2 to 1.7 @ 100 MHz, based on theoretical calculations. The dielectric constant of a composite will depend on the matrix material and volume loading of glass bubbles.

Scotchlite™ Glass Bubbles
K Series

Typical Properties *(continued)*

Particle Size: (See Product Specifications, paragraph D for size specification)

	Particle Size (microns, by volume)			
	Distribution			Effective top size
	10^m%	50^m%	90^m%	
K1 Glass Bubbles	30	65	110	120
K15 Glass Bubbles	30	60	105	115
K20 Glass Bubbles	30	65	110	120
K25 Glass Bubbles	25	55	95	105
K37 Glass Bubbles	20	40	80	85
K46 Glass Bubbles	15	40	70	80

Appearance: White to the unaided eye.

- 3 -

Scotchlite™ Glass Bubbles
K Series

Product Specifications

This specification covers hollow, unicellular glass microspheres, hereafter referred to as Glass Bubbles. Glass Bubbles are composed of a water resistant and chemically stable soda-lime-borosilicate glass.

Requirements:

A representative sample of Glass Bubbles will conform to the following requirements:

A. Isostatic Crush Strength:

Product	Test Pressure psi	Target % Survival	Minimum % Survival
K1	250	90%	80%
K15	300	90%	80%
K20	500	90%	80%
K25	750	90%	80%
K37	3,000	90%	80%
K46	6,000	90%	80%

Test Method: 3M TM-2028

B. Density:

Product	True Density (g/cc) Typical	Minimum	Maximum
K1	0.125	0.10	0.14
K15	0.15	0.13	0.17
K20	0.20	0.18	0.22
K25	0.25	0.23	0.27
K37	0.37	0.34	0.40
K46	0.46	0.43	0.49

Test Method: ASTM D2840*

*Sampling – In order to obtain representative samples of Glass Bubbles for density measurement via ASTM D2840, use 3M's vacuum sampling procedure or equivalent to avoid breakage. 3M certified density values are obtained with continuous in-line sampling equipment which does not cause product breakage.

C. Size:

1. Hard Particles – no particles (e.g. glass slag, flow agent, etc.) greater than U.S. number 40 (420 microns) standard sieve will exist.

2. Oversize Particles –
   For K1, K15, K20 and K25 Glass Bubbles:
   When tested in accordance with ASTM D1214, using a 10 gram sample on a U.S. Number 80 standard sieve (177 microns), a maximum of five (5) percent by weight Glass Bubbles will be retained on the sieve.

Scotchlite™ Glass Bubbles
K Series

Product Specifications *(continued)*

2. Oversize Particles – *(continued)*
   For K37 and K46 Glass Bubbles:
   When tested in accordance with ASTM D1214, using a 10 gram sample on a U.S. Number 100 standard sieve (149 microns), a maximum of one (1) percent by weight Glass Bubbles will be retained on the sieve.

D. Alkalinity:

Maximum of 0.5 milliequivalents per gram

Test Method: ASTM D3100 (1982 edition)

E. Flotation:

| | Floaters (% by bulk volume) | |
Product	Typical	Minimum
K1	96%	90%
K15	96%	90%
K20	96%	90%
K25	96%	90%
K37	94%	90%
K46	92%	90%

Test Method: 3M TM-588

F. Volatile Content:

Maximum of 0.5 percent by weight.

Test Method: 3M TM-587

G. Flow:

Scotchlite™ Glass Bubbles remain free flowing for at least one year from the date of shipment if stored in the original, unopened container in the minimum storage conditions of an unheated warehouse.

H. Packaging:

Glass Bubbles will be packaged in suitable containers to help prevent damage during normal handling and shipping. Each container will be labeled with:

1. Name of manufacturer
2. Type of Glass Bubbles
3. Lot number
4. Quantity in pounds

For further information on properties not covered in this specification, refer to Typical Properties.

Scotchlite™ Glass Bubbles
K Series

Storage and Handling

To help ensure ease of storage and handling while maintaining free flowing properties, Scotchlite™ Glass Bubbles have been made from a chemically stable glass and are packaged in a heavy duty polyethylene bag within a cardboard container.

Storage:

Minimum storage conditions should be unopened cartons in an unheated warehouse.

Under high humidity conditions with the ambient temperature cycling over a wide range, moisture can be drawn into the bag as the temperature drops and the air contracts. The result may be moisture condensation within the bag. Extended exposure to these conditions may result in "caking" of the Glass Bubbles to various degrees. To minimize the potential for "caking" and prolong the storage life, the following suggestions are made:

1. Carefully re-tie opened bags immediately after use.
2. If the polyethylene bag is punctured during shipping or handling, use this bag as soon as possible, patch the hole, or insert the contents into an undamaged bag.
3. During humid summer months, store in the driest, coolest space available.
4. If good storage conditions are unavailable, carry a minimum inventory, and process on a first in/first out basis.

Handling:

Dusting problems that may occur while handling and processing can be minimized by the following procedures:

1. For eye protection wear chemical safety goggles. For respiratory system protection wear an appropriate NIOSH/MSHA approved respirator. (For additional information about personal protective equipment, refer to Material Safety Data Sheet.)
2. Use appropriate ventilation in the work area.
3. Pneumatic conveyor systems have been used successfully to transport Glass Bubbles without dusting from shipping containers to batch mixing equipment. Static eliminators should be used to help prevent static charges.

   Diaphragm pumps have been used successfully to convey Glass Bubbles. Vendors should be consulted for specific recommendations.

Glass Bubble Breakage:

Glass bubble breakage may occur if the product is improperly processed. To minimize breakage, avoid high shear processes such as high speed Cowels Dissolvers, point contact shear such as gear pumps or 3-roll mills, and processing pressures above the strength test pressure for each product.

Health and Safety Information

For product Health and Safety Information, refer to product label and Material Safety Data Sheet (MSDS) before using product.

Scotchlite™ Glass Bubbles
K Series

Packaging Information

Large Box (50 cubic ft.)

Description: A single corrugated box with a plastic liner. Top enclosed with interlocking double cover banded. Bottom is normal box closure, entire box banded to wooden pallet.

Dimensions: Box I.D. of 48 in. x 42 in. x 44 in. overall load size is 48³/₄ in. x 42³/₄ in. x 50 in. including pallet. Pallet size is 42 in. x 48 in.

Small Box (10 cubic ft.)

Description: A single corrugated box with a plastic liner. All boxes are banded together and to the wooden pallet. 4 Boxes per pallet.

Dimensions: Each box I.D. is 22 in. x 19 in. x 39 in. Pallet size is 42 in. x 48 in.

Box Weights

Product	Small Box	Large Box*	Truckload Large Box* 44 pallets
K1	40 lb.	210 lb.	9,240 lb.
K15	50 lb.	265 lb.	11,660 lb.
K20	60 lb.	350 lb.	15,400 lb.
K25	80 lb.	430 lb.	18,920 lb.
K37	100 lb.	660 lb.	29,040 lb.
K46	125 lb.	815 lb.	35,860 lb.

*Box weights may vary due to manufacturing tolerances on each product.

Scotchlite™ Glass Bubbles
K Series

For Additional Information or Sales Assistance	3M Specialty Additives 1-800-367-8905 • Fax 612-736-4133 In Canada, 1-800-410-6880, Ext. 6019 In Puerto Rico, 809-750-3000.
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SELECTED BIBLIOGRAPHY

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12.0 INDEX

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12.0 INDEX

Wettability: 56, 88, 93, 94
Working time: 42, 47, 92
Zone: 13, 23-25, 27, 29, 30, 31, 35